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Dr. Rock

ANNUAL REPORT

to

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

for

CHARACTERIZATION OF THE PHYSICO-CHEMICAL PROPERTIES OF POLYMERIC MATERIALS FOR AEROSPACE FLIGHT

NSG-5009

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ABSTRACT

The differential thermal analyzer is a very suitable instrument for the rapid analytical study of the thermal behavior of battery electrodes. Solid samples can be studied in the range of $0^{o}C$-$500^{o}C$ using the standard cell assembly. Thermal behavior of the battery electrodes is automatically recorded by the analyzer and it can be used for qualitative analysis. A study is also being made of the behavior of battery electrodes which have been charged at different levels.

INTRODUCTION

Differential thermal analyses are conducted with a DuPont Model 900 DTA unit. DTA is a technique for studying the thermal behavior of materials as they undergo physical and chemical changes during heating and cooling. The 4mm-diameter tubes, one containing sample and the other containing a reference material, such as glass beads, are heated at a uniform rate in a heating block. The temperature differential between the two tubes will remain zero as they are heated unless the sample undergoes an endothermic or exothermic reaction. A thermocouple is inserted in the tube containing the sample and another thermocouple is inserted in the tube containing the glass beads. The glass beads do not undergo any chemical change in the temperature range under study. As long as the temperature of the sample equals the temperature of reference material, the two thermocouples produce identical voltage and the net voltage differential is zero. When an exothermic or an endothermic change takes place in the sample, the sample temperature no longer equals the reference temperature and the resultant voltage differential reflects the difference in temperature and either a positive or negative ΔT peak on the graph results. The DTA unit plots the temperature of the heating block on the X-axis; on the Y-axis it plots the difference in temperature between the sample and the reference, ΔT. An exotherm is plotted as a rise from the base line; an endotherm as a decrease from the base line.

DISCUSSION OF THE RESULTS

Several positive and negative battery electrodes were analyzed. The negative plates show a first endotherm between 245°C and 250°C. This is a very large peak. The second endotherm occurs at 300°C which is indicative of the decomposition of $Cd(OH)_2$ (see graph 1 to 6). In the analysis of positive plates, a first weak endotherm occurs at 100°C, which indicates loss of H_2O from $Ni(OH)_2(H_2O)_n$. A second large endotherm occurs in the range of 290°C-300°C, which is indicative of the decomposition of $Ni(OH_2)$ to NiO and H_2O (see graphs 7 - 17).

ABSTRACT

Atomic Absorption Spectroscopy is used to determine nickel, cobalt, cadmium, and potassium content in battery electrolytes and electrodes. We are also determining the interference effects of one element in the presence of others. Atomic Absorption is a quick and accurate method for the determination of traces of the above mentioned metals.

INTRODUCTION

Sealed Ni-Cd cells have proved to be a useful and reliable rechargeable source of power for aerospace applications. However, it has been found that sometimes these cells have failed.

Although it is not completely known what leads to such failures, it has been found experimently that some of the factors which contribute to the final failure of the batteries are:

1. Extent and nature of cycle regime
2. Operating temperature
3. Carbonate contamination
4. Cd migration
5. Nature and condition of separator

The analysis of negative electrodes, positive electrodes, and of the electrolyte is also important.

A.A spectroscopy is being used to analyze the elements of interest (Ni, Cd, Co, and K) in the electrodes and electrolytes of the Ni-Cd cells.

These results have been compared with those obtained by standard chemical analysis method and are in agreement. A.A spectroscopy is much quicker and embraces virtually all alloying components contained in Ni-Cd cells.

This method is being used to analyze for concentration of trace metals in negative and positive electrodes of batteries. This should prove useful in determining the amount and effects of these trace metals in functioning and durability of Ni-Cd cells.

During the second half of the year, three (3) new students worked on the project and received research training and experience both at GSFC and Bowie State College.

Most of the work during this period was on battery electodes. The data were collected in conjunction with Dr. K. Vasanth, at GSFC, and the results are interpreted in Tables XXI and XXII.

The Atomic Absorption Spectrophotomer at Bowie State College was checked against the unit at GSFC and the results were in agreement.

INSTRUMENTATION

A Perkin-Elmer Model 403 Atomic Absorption Spectrophotometer was used at Goddard Space Flight Center. This unit has a digital read-out panel. High intensity cathode tubes for Ni, Cd., and Co were used depending on which element was being measured. Operating conditions were generally those recommended in the Analytical Methods Book.

The steps listed below were followed in adjusting the Model 403 Spectrophotometer in preparation for performing the analysis.

1. The instrument and exhaust hood are turned on and allowed warm-up at the specified current given in the Analytical Method Book for two hours or until stability is achieved. Stability is achieved when no zero shift is apparent over a five minute interval.

2. The air supply is turned on and the air pressure is set at 62 lbs/sq. in.

3. The acetylene supply is turned on and acetylene pressure is set at 27 lbs/sq. in.

4. The burner is ignited.

5. The flame should be blue and transparent with an oxidizing region about 4mm.

6. The slit control is set at the value given in the Analytical Methods Book for the respective elements.

7. The adjustment of the atomizer is made by turning the capillary outward until "blow-back" occurs, then, turning inward until absorption is maximized. Standard solutions are aspirated through a tube into the flame for not less than 15 seconds.

A Varian Model 1200 N.A. Spectrophotometer was used at Bowie State College.

KNOWN SOLUTIONS PREPARATION

The solutions used were prepared from standard solutions of 1000 (Parts per Million (PPM). The dilutions were made as follows:

10 ml of 1000 PPM standard solutions were diluted to a final volume of 500 ml with deionized water to give a solution of 20 PPM concentration. This 20 PPM solution was used as a stock solution. Further dilutions were made as follows:

1. 5 ml of 20 PPM solution was diluted with deionized water to give a final volume of 100 ml and a solution of 1 PPM.

2. 10 ml of 20 PPM solution was diluted to a final volume of 100 ml and a solution of 2 PPM.

3. Repeat the above procedure with 15 ml of stock solution to get 3 PPM solution.

4. Repeat the above procedure with 20 ml stock solution to yield a solution of 4 PPM.

5. Repeat above procedure with 25 ml of stock solution to get a solution of 5 PPM.

6. Repeat above procedure with 30 ml of stock solution to get a solution of 6 PPM.

7. Repeat above procedure with 35 ml of stock solution to get a solution of 7 PPM.

8. Repeat above procedure with 40 ml of stock solution to get a solution of 8 PPM.

9. Repeat above procedure with 45 ml of stock solution to get a solution of 9 PPM.

10. 50 ml of stock solution are diluted with 50 ml deionized water to get a final solution of 10 PPM.

DRAWING OF CALIBRATION CURVE

The Atomic Absorption Spectrophotometer readings are displayed in absorption but they can be readily converted by means of a table to percent absorption which varies almost linearly with concentration. The conversion table is provided in the Analytical Methods Book for the Perkins-Elmer Model 403 A.A Spectrophotometer.

The instrument parameters are recorded with each set of data so they can be duplicated when corresponding sample runs are made. Each curve standard is run in ascending order of element concentration. Curves can be conveniently plotted on expanded logrithmic paper.

ANALYSIS OF SAMPLES

The agreement of the results obtained by A.A. Spectroscopic analyses with those obtained by standard analyses have previously been confirmed. (Please see annual report 1979.)

For analysis of each sample a calibration curve is derived from standard solutions. The given samples are diluted and the concentration of metal in the aliquot is calculated from the calibration curve. This is multiplied by the dilution factor to give the concentration of the metal in the original sample.

The results obtained are given in Table Ia through Table XXb. Tables "a" contain the data for the standard calibration curve and tables "b" contain the data for analyzed samples.

The point corresponding to each analyzed sample has been marked on the calibration curve.

Analyses of the electrodes were made according to Procedures for Analysis of Nickel-Cadmium Cell Materials by Holpert, G., Webster, W.H., Jones, C.C., and Ogunyankin, O., GSFC Publication X-711-74-279, October 1974. Tables XXI and XXII give results of analyses of positive and negative battery plates.

In Table XXI

Column 1 - The group number of the design variable cells

Column 2 - The serial number of the cell

Column 3 - The pack number is assigned to the cells when they are cycled at the Naval Weapons Center, Crane, Indiana. Uncycled cells have no pack number.

Column 4 - The number of charge/discharge cycles which the cell has been subjected to.

Column 5 & 6 - The average thickness of the plate to the nearest 0.1mm. This is determined by measuring the plate at the top, middle and bottom.

Columns 7 & 8 - The weight of each plate to the nearest 0.01g.

In Table XXII

Column 1 - The Group-Serial number of the cell

Column 2 - The theoretical capacity of the positive electrode as calculated from the amount of active nickel, Ni(II) ion plus Ni(IV) ion in the electrode.

The Ni(IV) may be determined by reducing it with a known excess of Fe(II) and titrating the excess Fe(II) with standard permanganate. The total active nickel may be determined by titration with EDTA.

The total theoretical amp-hr is then calculated from the number of equivalents of active nickel.

Column 3 - The theoretical capacity of the negative electode as calculated from the total active cadmium, Cd plus Cd(II) in the electrode.

The Cd(II) is titrated with EDTA. The Cd is converted to Cd(II) separated from iron and nickel and titrated with EDTA.

The total theoretical amp-hrs is calculated from the number of equivalents of cadmium.

Columns 4 & 5 - The actual plate capacity as measured electrochemically. A comparison of electochemical capacity and chemical capacity gives a measure of the unavailable material in the electrode.

Column 6 & 7 - The milliequivalents of hydroxide and carbonate ions in the electrolyte.

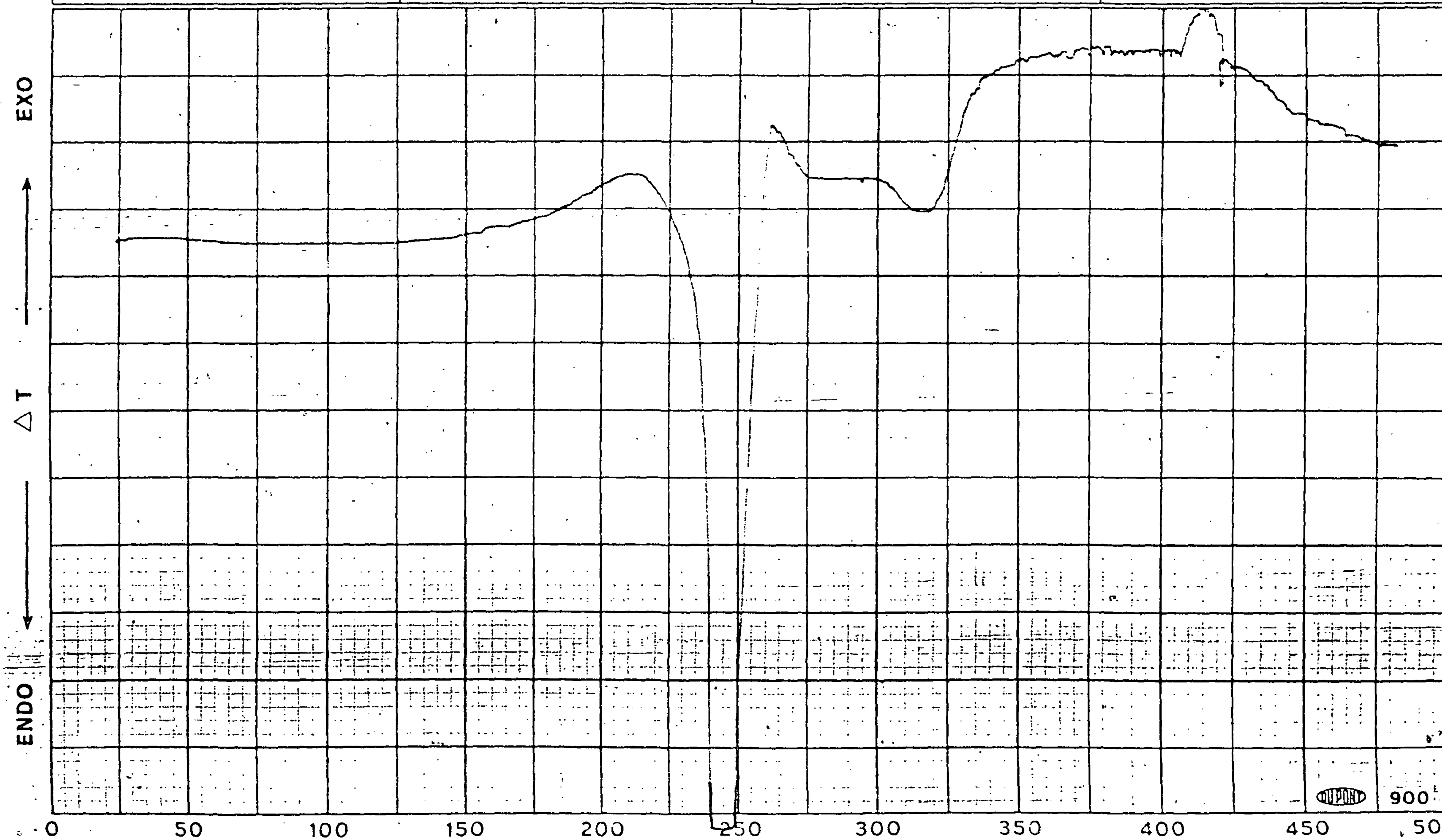
Column 8 - The percent cobalt in the positive plate. This is determined by A.A. spectroscopy.

EXPERIMENTAL - PART I

GRAPHS OF DIFFERENTIAL

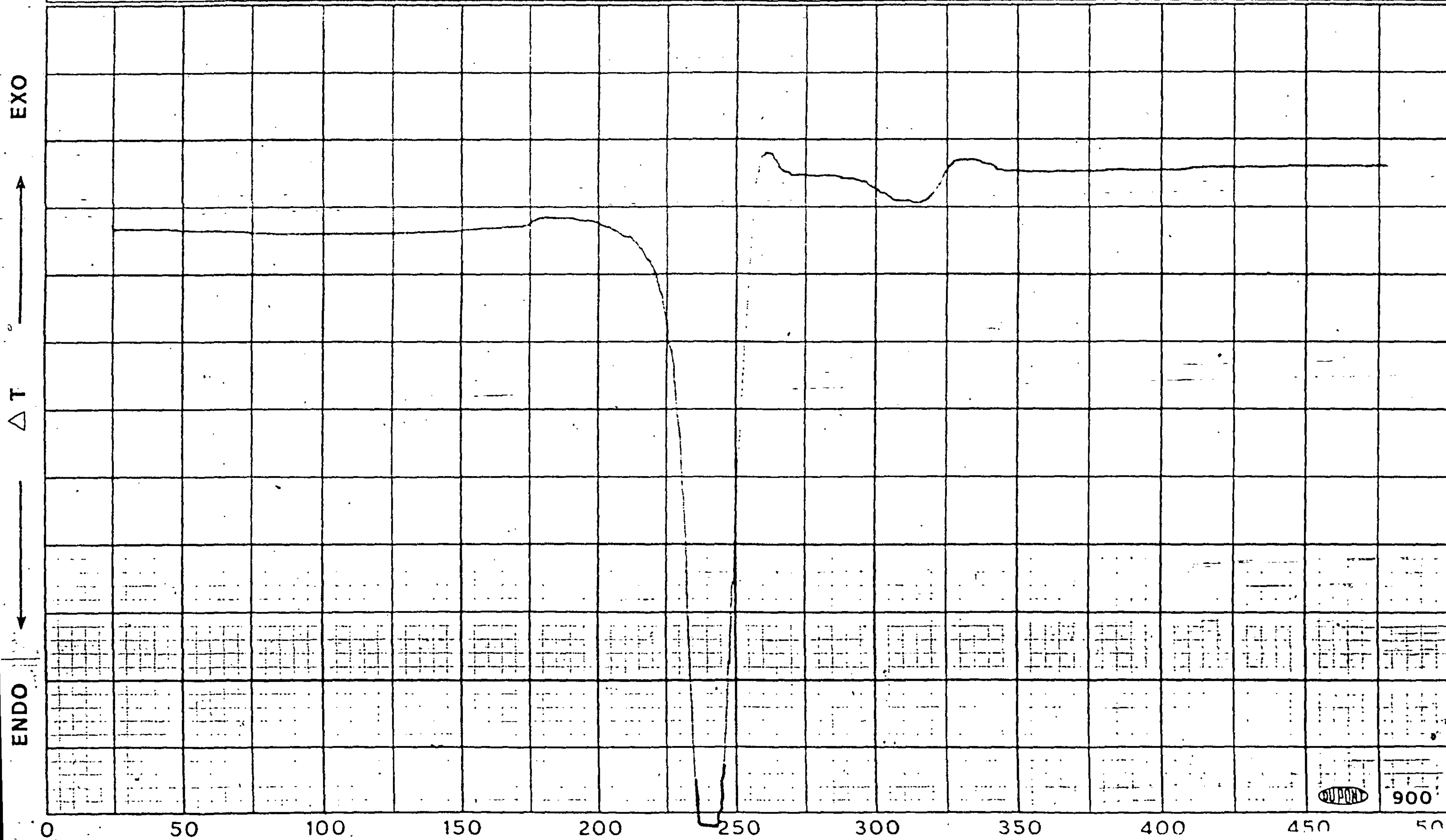
THERMAL ANALYSES

SAMPLE:	SIZE	ATM.	RUN NO.
	REF.	SCALE SETTING — T: 50 °C/IN.; △T: 1.0 °C/IN.	DATE
	PROGRAM MODE		OPERATOR
ORIGIN:	RATE 6 °C/MIN, START °C		

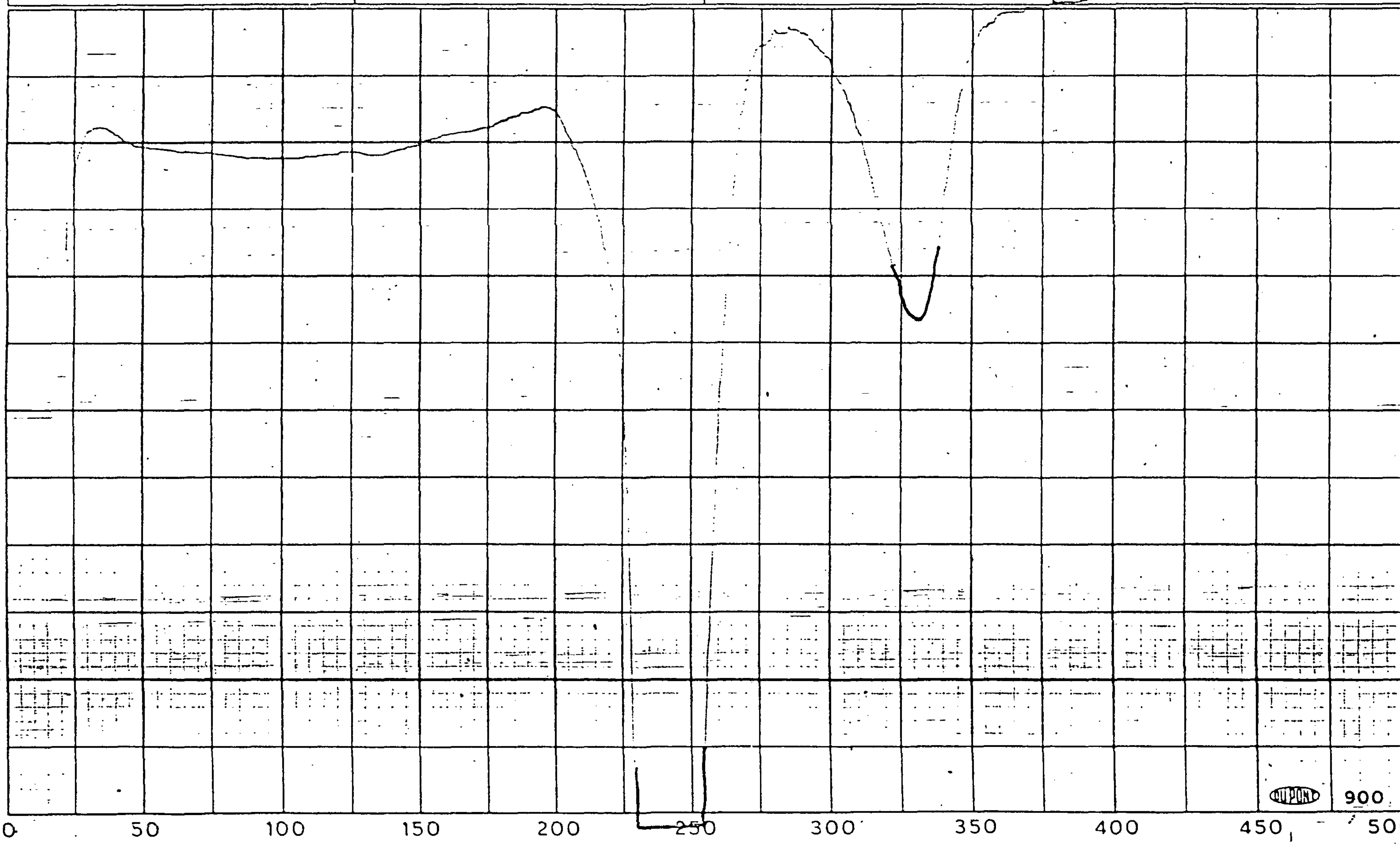


#2

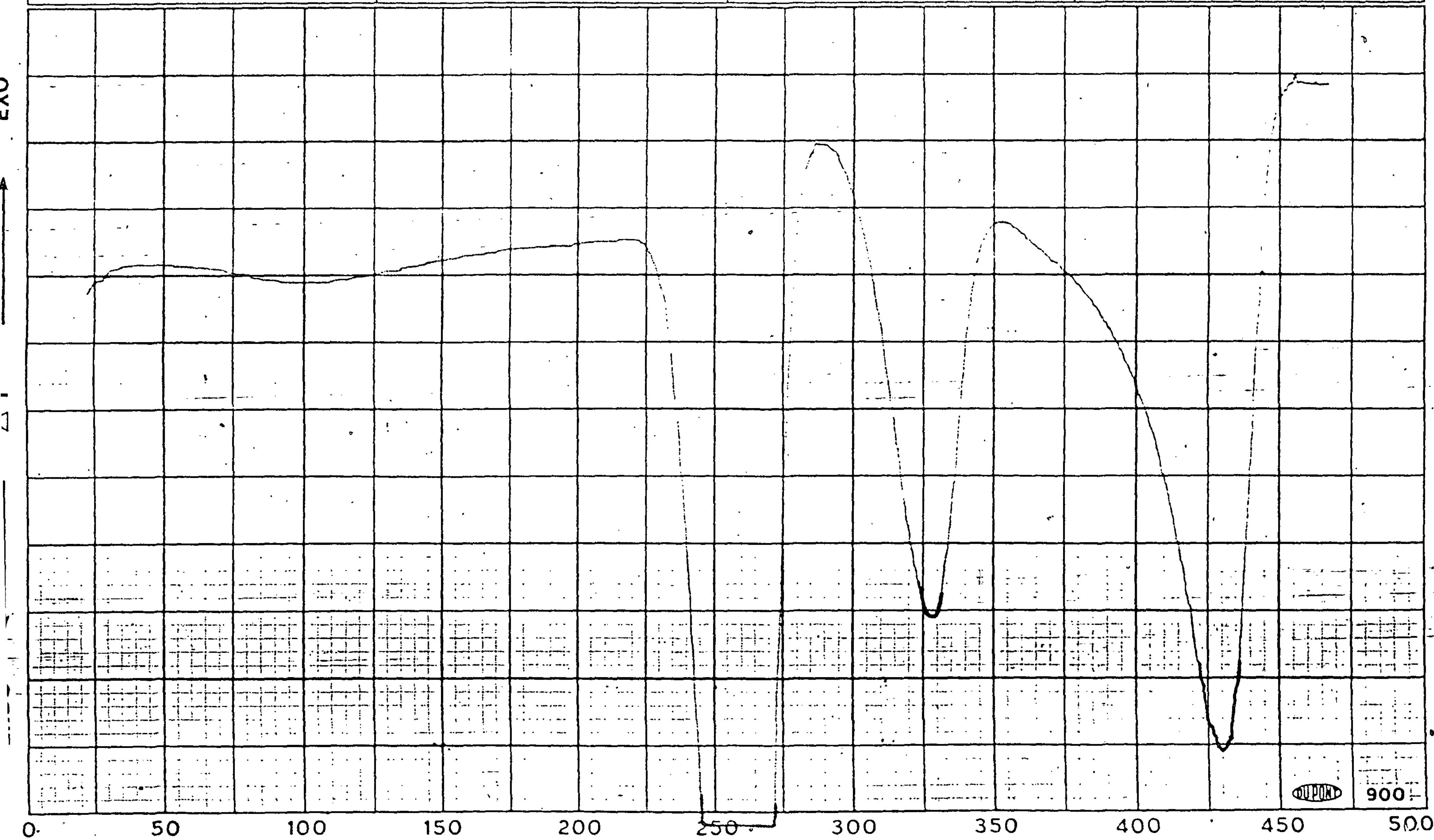
SAMPLE:	SIZE	ATM.	RUN NO. 10
	REF.		DATE
	PROGRAM MODE	SCALE SETTING: T 50 °C/IN. ΔT 1 °C/IN.	OPERATOR
ORIGIN:	RATE °C/MIN, START °C		



SAMPLE: EP SN 202 Mrj #5	SIZE 2mm in depth	ATM.	RUN NO. #3
ORIGIN: [illegible]	REF. glass Beads	SCALE SETTING: T 50 °C/IN.; ΔT 1 °C/IN.	DATE 6.24.80
	PROGRAM MODE heat		OPERATOR S. Khan
	RATE 15 °C/MIN. START 85 °C		



SAMPLE:	SIZE	ATM.	RUN NO. #4
	REF.	T / △T	DATE
	PROGRAM MODE	SCALE SETTING 50 °C/in.	OPERATOR
ORIGIN:	RATE °C/min. START °C		



DUPONT INSTRUMENT PRODUCTS DIVISION

5

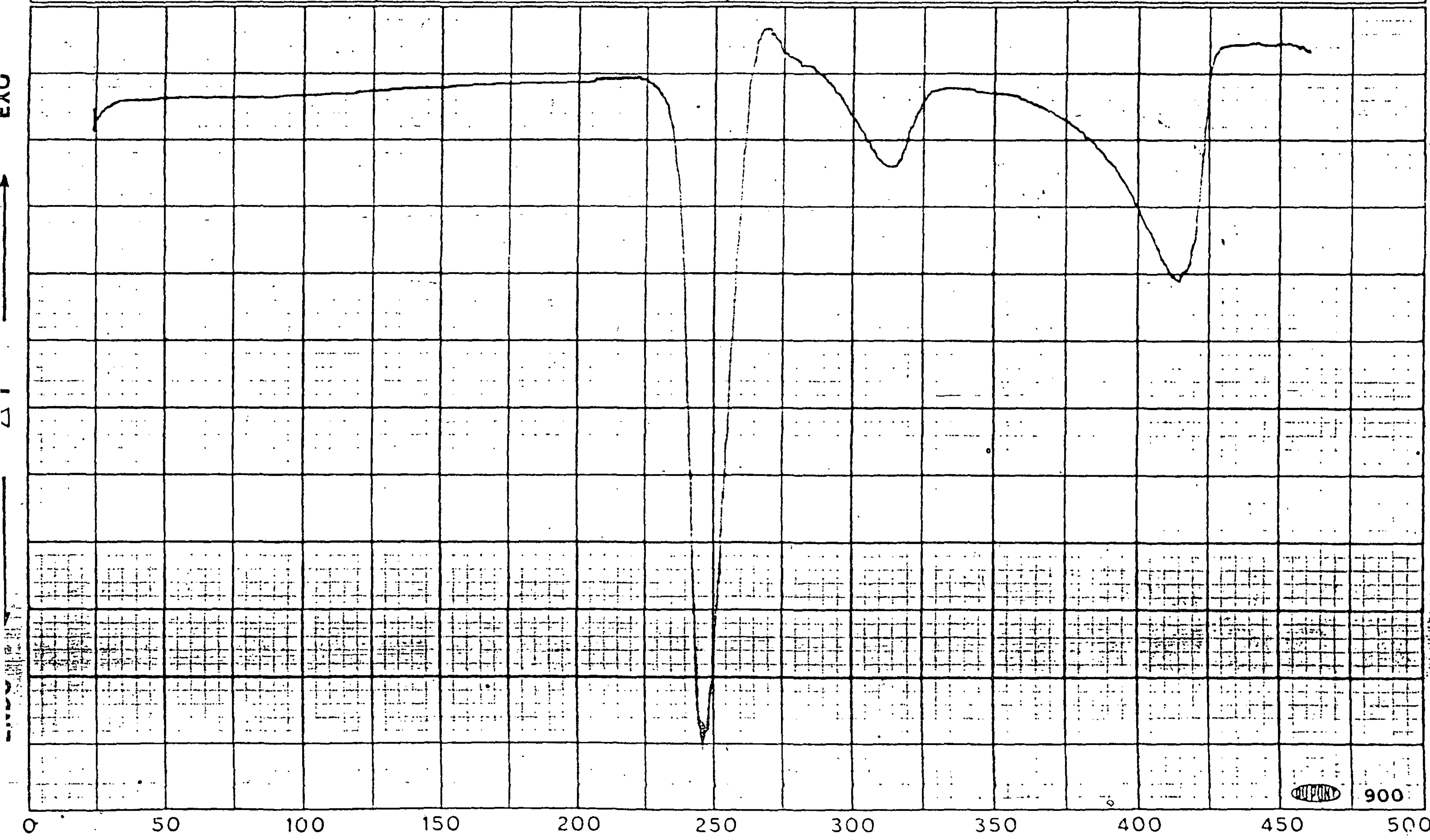
SAMPLE: Neg Plate
Lot NO. 555 H
Part No. 805040
ORIGIN:

SIZE 8 mm in depth
REF. glass leads
PROGRAM MODE heat
RATE 7.0 °C/MIN, START Room °C 24

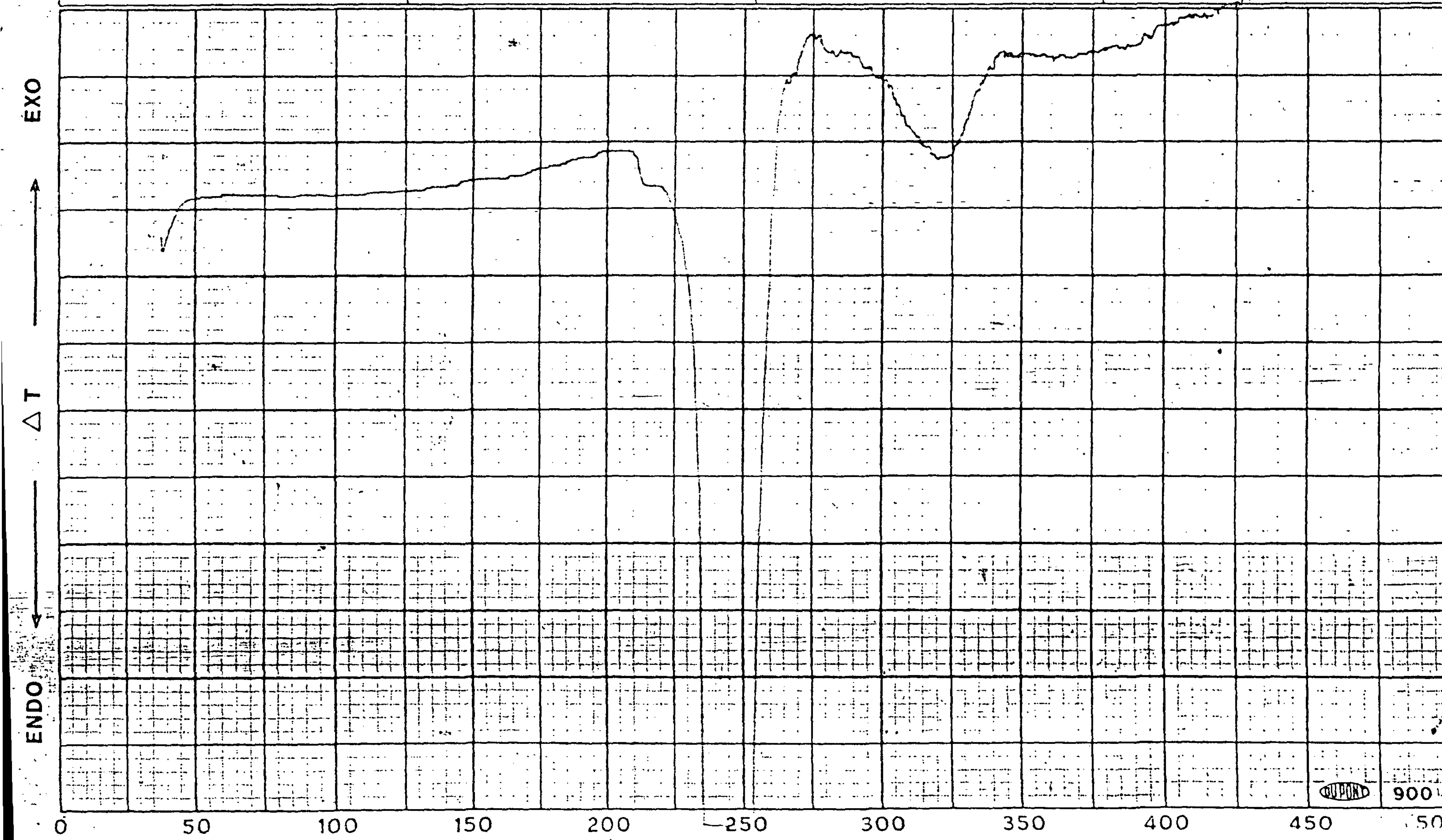
ATM.

	T	ΔT
SCALE SETTING	50 °C/IN.	1.0 °C/IN.

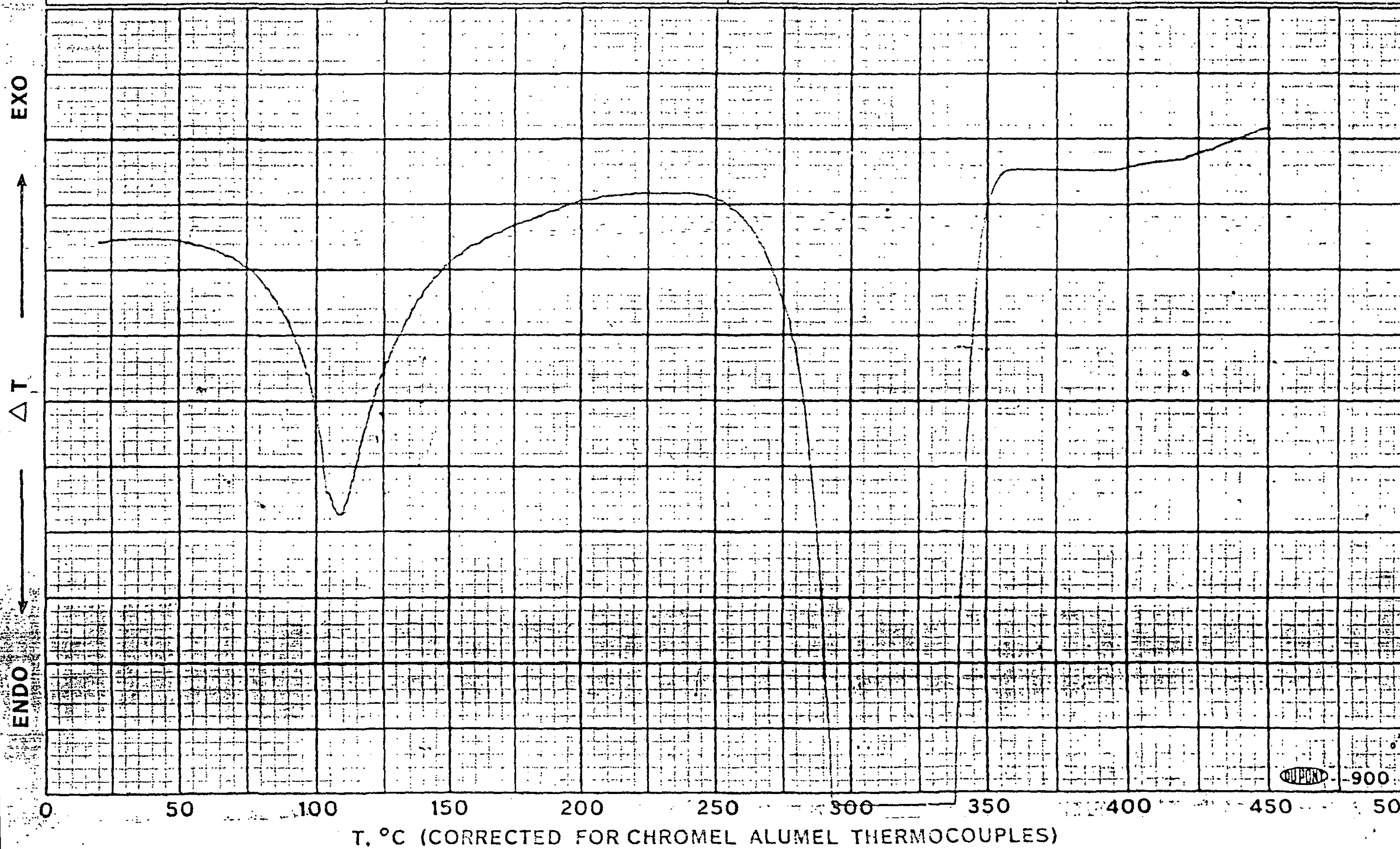
RUN NO. 14
DATE 6.30.80
OPERATOR S. Khan



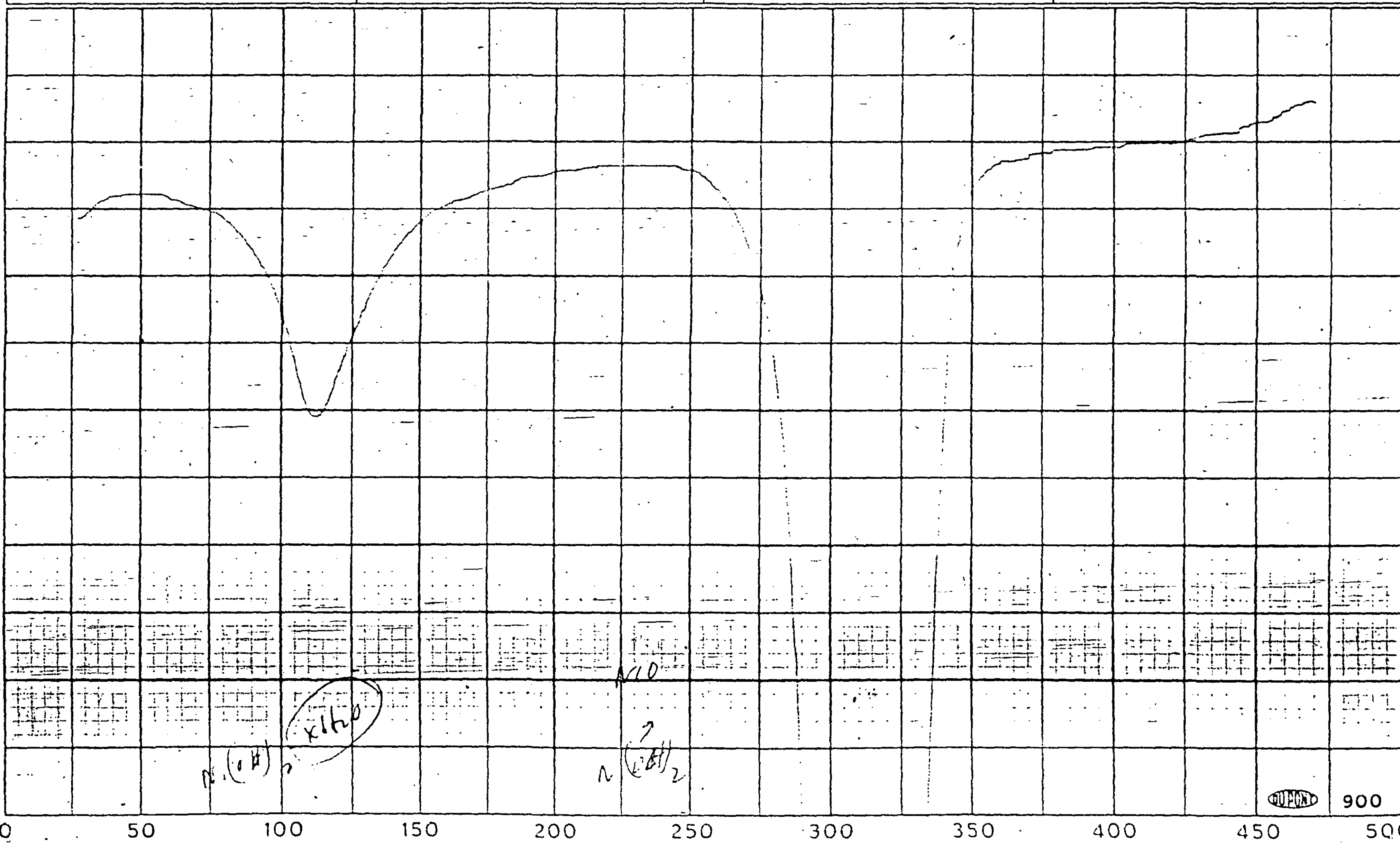
SAMPLE: GE 001 veg. Blak #17 Lot #2 ORIGIN:	SIZE ___ REF. glass beads PROGRAM MODE heat RATE 10 °C/MIN, START 25°C	ATM. SCALE SETTING: T 50 °C/IN., ΔT 1 °C/IN.	RUN NO. #6 DATE 6-25-80 OPERATOR C.Klam



SAMPLE: EP - SN 202	SIZE thin deck	ATM.	RUN NO. 1
Pos #2	REF. glass beads	T: — ; ΔT: —	DATE 6.24.80
	PROGRAM MODE heat	SCALE SETTING: T 50 °C/IN. ; ΔT 1 °C/IN.	OPERATOR S. Khan.
ORIGIN: Dr. Vasant	RATE 15 °C/MIN, START 24 °C		



SAMPLE: EP-SN 202 -Pos #12	SIZE 2 mm in depth	ATM.	RUN NO. 2
	REF.		DATE 6-24-80
	PROGRAM MODE P...	SCALE SETTING T 50 °C/IN. ΔT 1 °C/IN.	OPERATOR E. Khan
ORIGIN: Dri-Vasait.	RATE 15 °C/MIN, START 24 °C		



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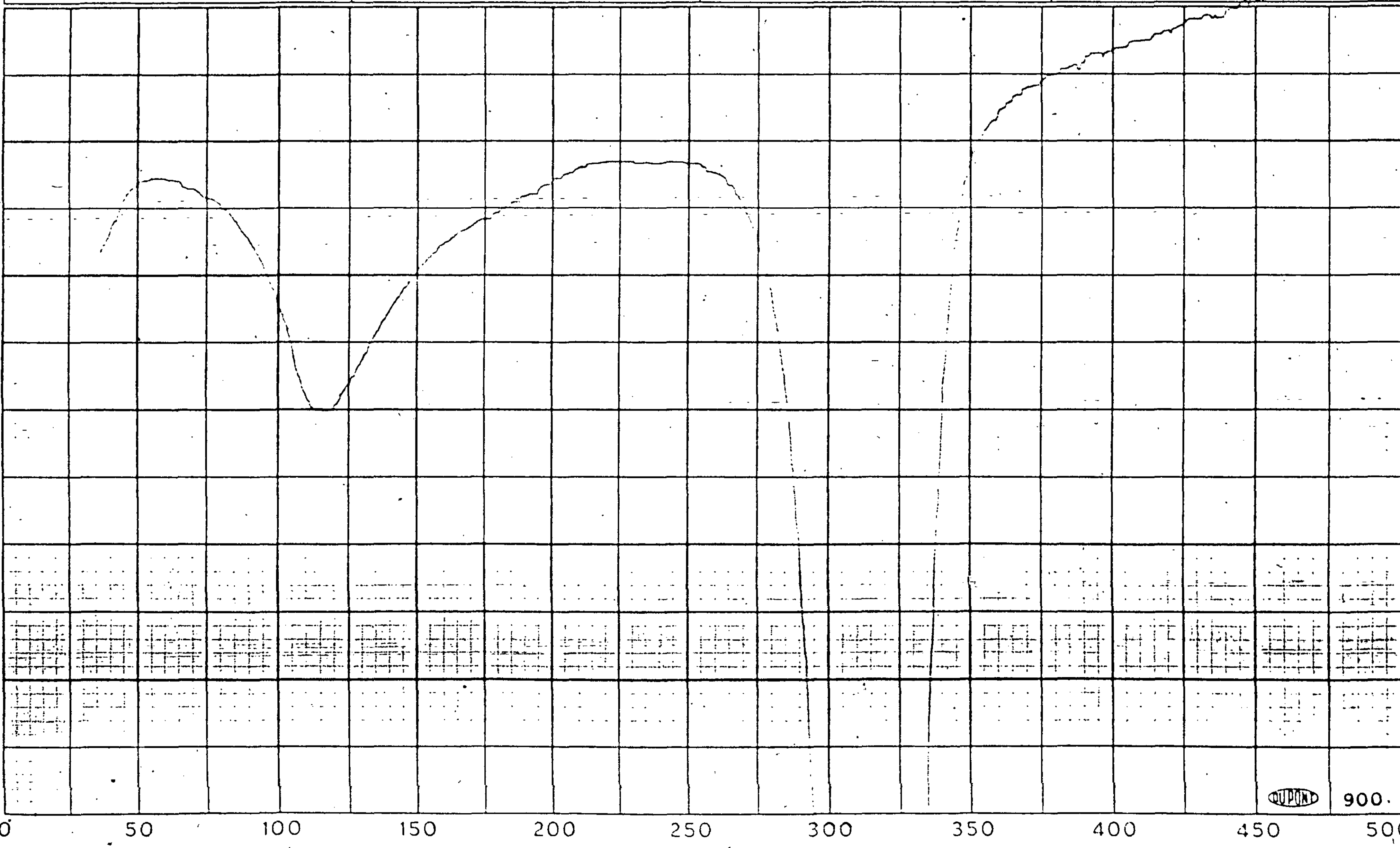
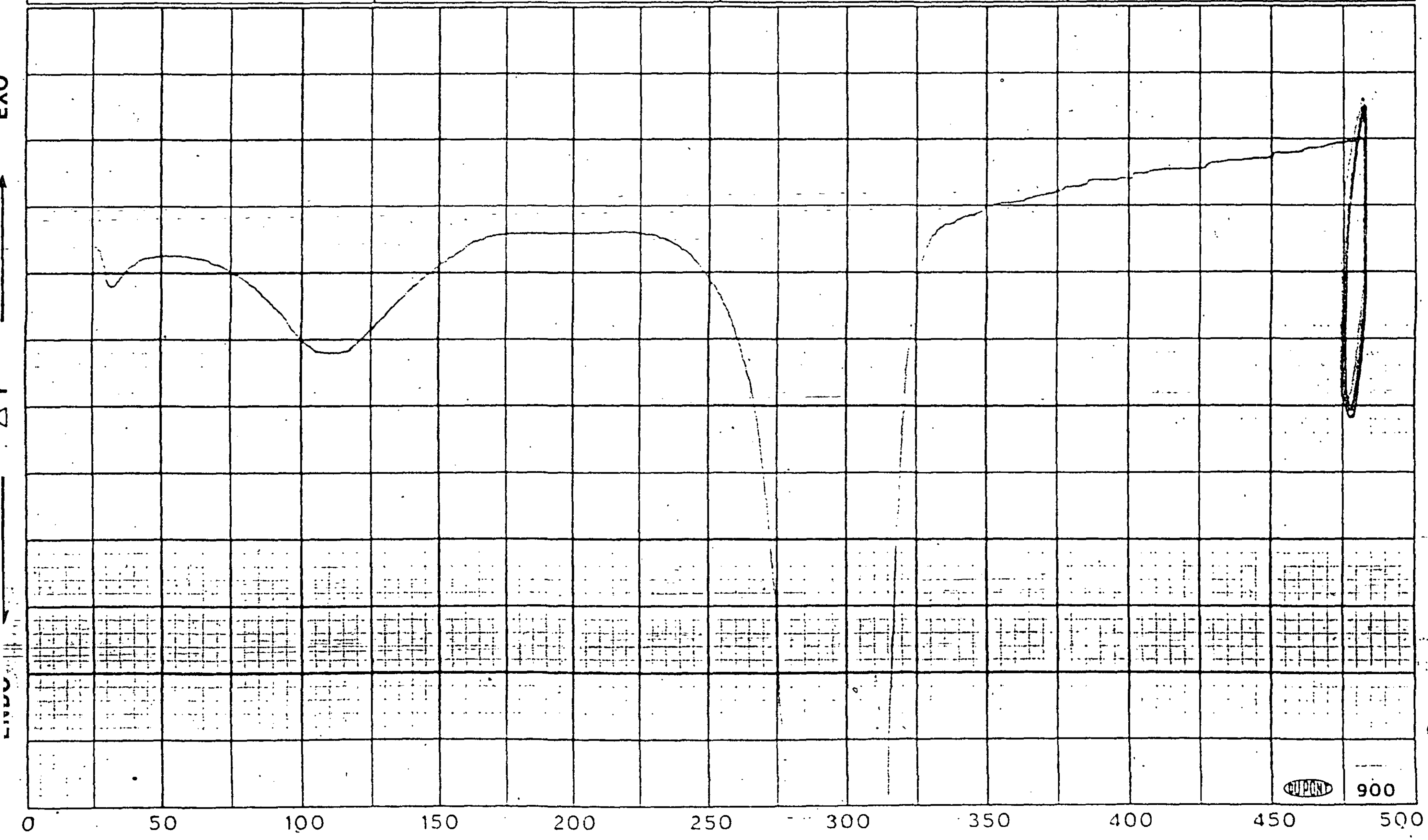
SAMPLE: Bs Plates Lot No. 553L Part No. 805027 ORIGIN: Diversified	SIZE [illegible] REF. glass Beads PROGRAM MODE [illegible] RATE 20 °C/MIN, START 25 °C	ATM. SCALE SETTING: T 50 °C/IN.; ΔT 1.0 °C/IN.	RUN NO. #5 DATE 6·25·80 OPERATOR [illegible]



SAMPLE: PoC GE 001 Lot [illegible]	SIZE 2 mm [illegible]	ATM.	RUN NO. #7
	REF. [illegible]	T / ΔT	DATE 6-26-[illegible]
	PROGRAM MODE [illegible]	SCALE SETTING 50 °C/IN. / 1.0 °C/IN.	OPERATOR [illegible]
ORIGIN:	RATE 15 °C/MIN, START 25 °C		



DU PONT INSTRUMENT PRODUCTS DIVISION

SAMPLE: TDSS 1

ORIGIN: [illegible]

SIZE: 3 [illegible]

REF.: Glass beads

PROGRAM MODE: heat

RATE: 15 °C/MIN, START: 20 °C

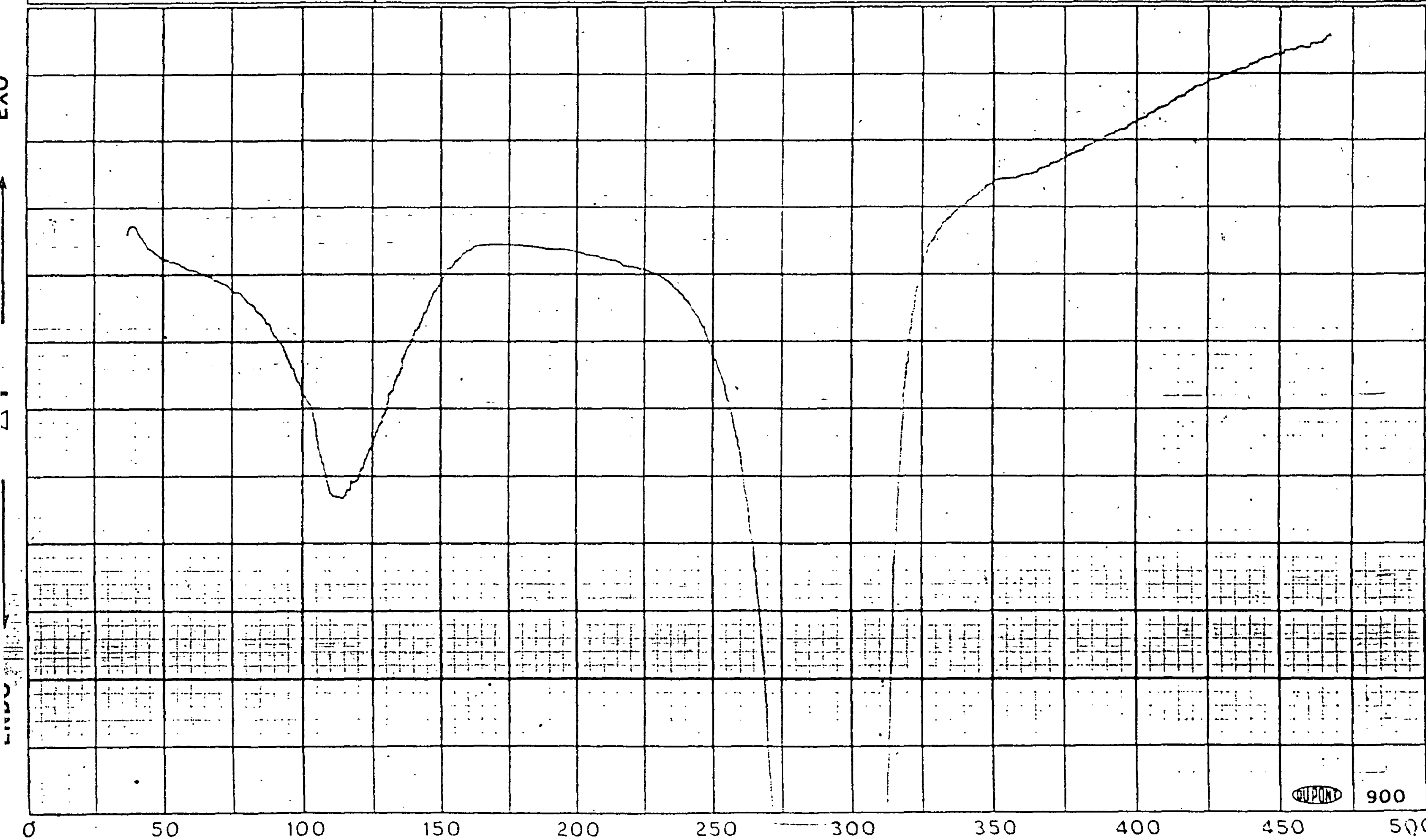
ATM.:

SCALE SETTING	T	ΔT
	50 °C/IN.	1.0 °C/IN.

RUN NO.: 8

DATE: 6.26.80

OPERATOR: C. Khan



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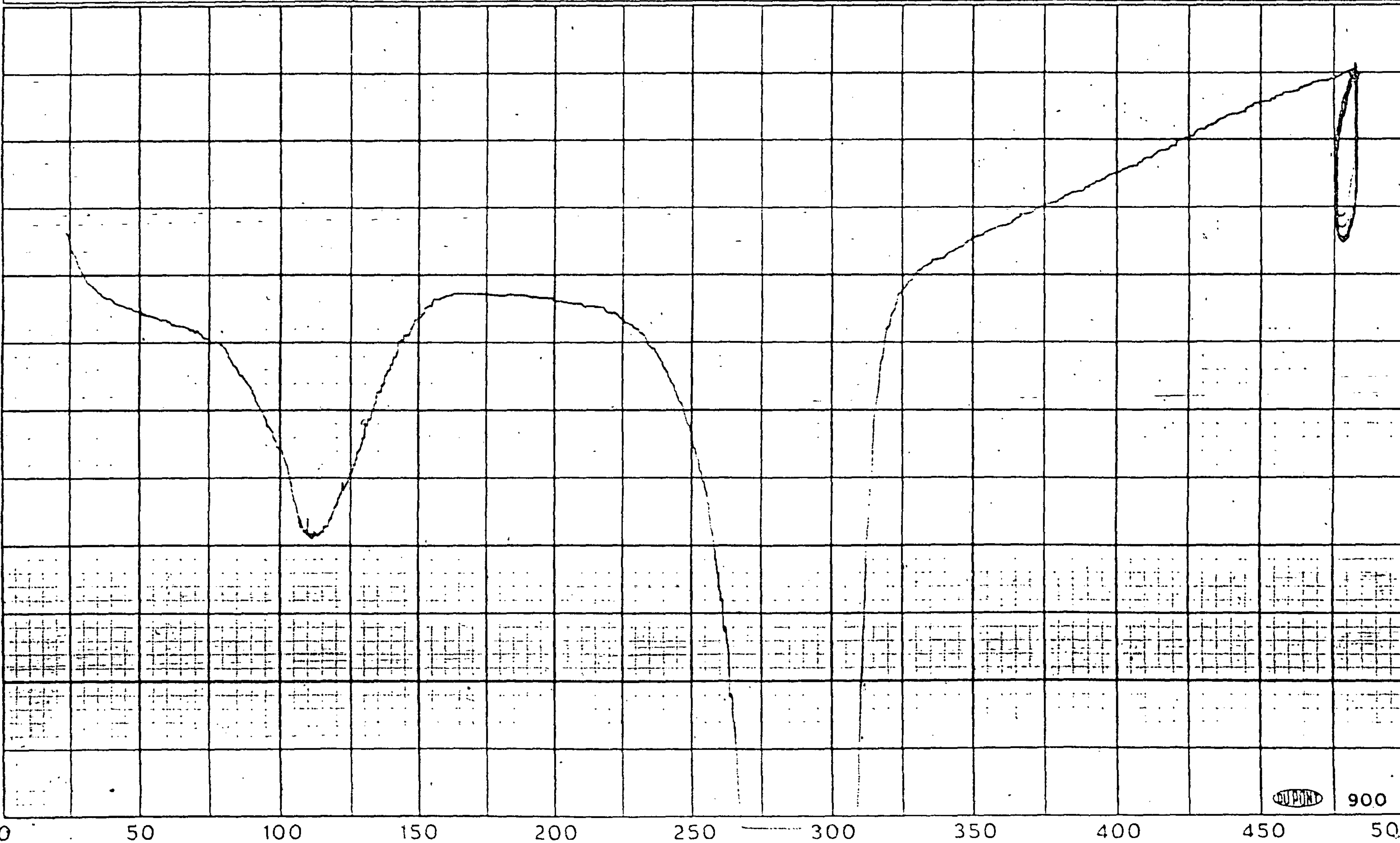
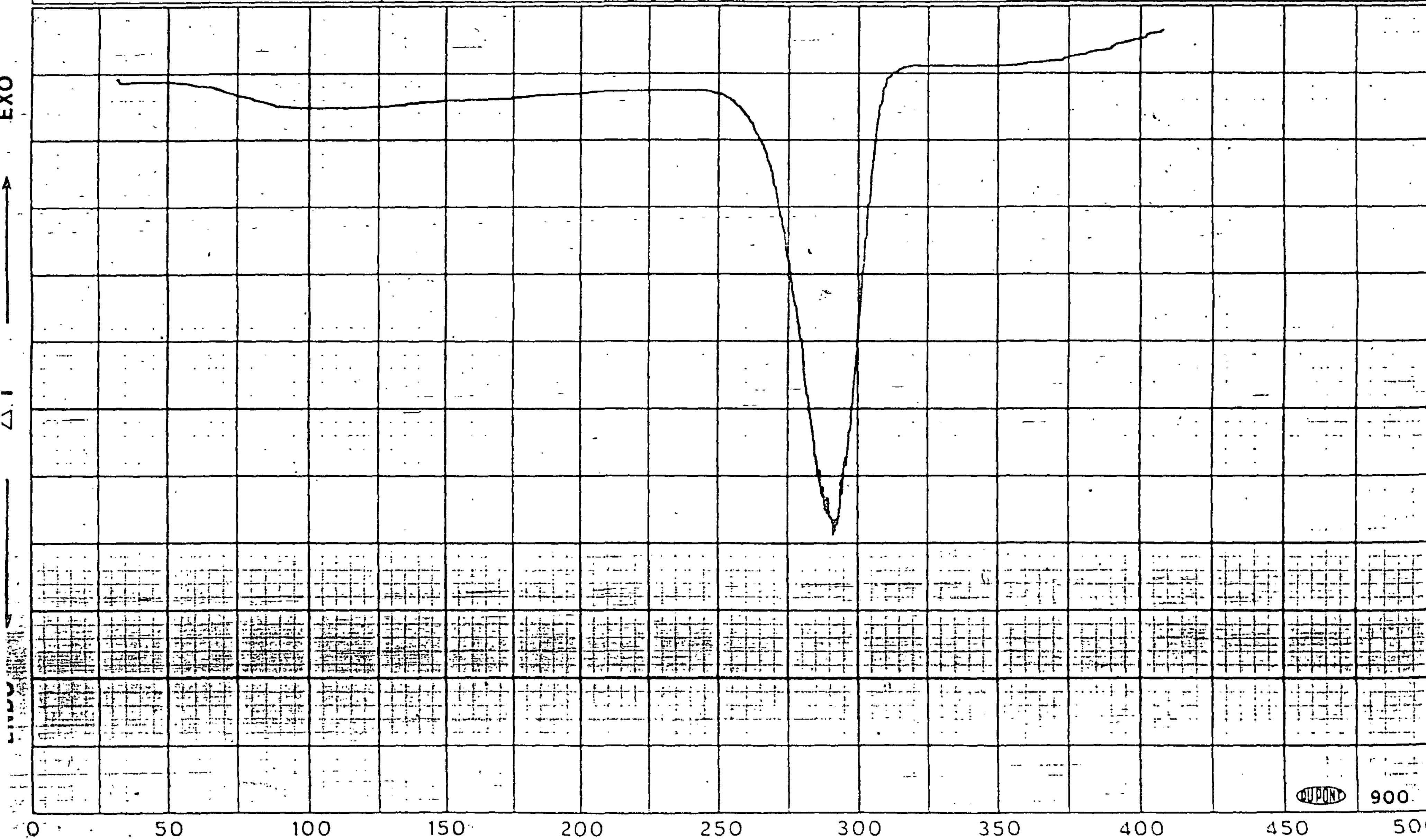
12

SAMPLE: TDRS 2	SIZE 3mm in depth	ATM.	RUN NO. 9
	REF. glass beads	T / ΔT	DATE 6 26 80
	PROGRAM MODE heat	SCALE SETTING 50 °C/IN. / 1.0 °C/IN.	OPERATOR S Klaus
ORIGIN: Dr. ...	RATE 15 °C/MIN, START 25 °C		

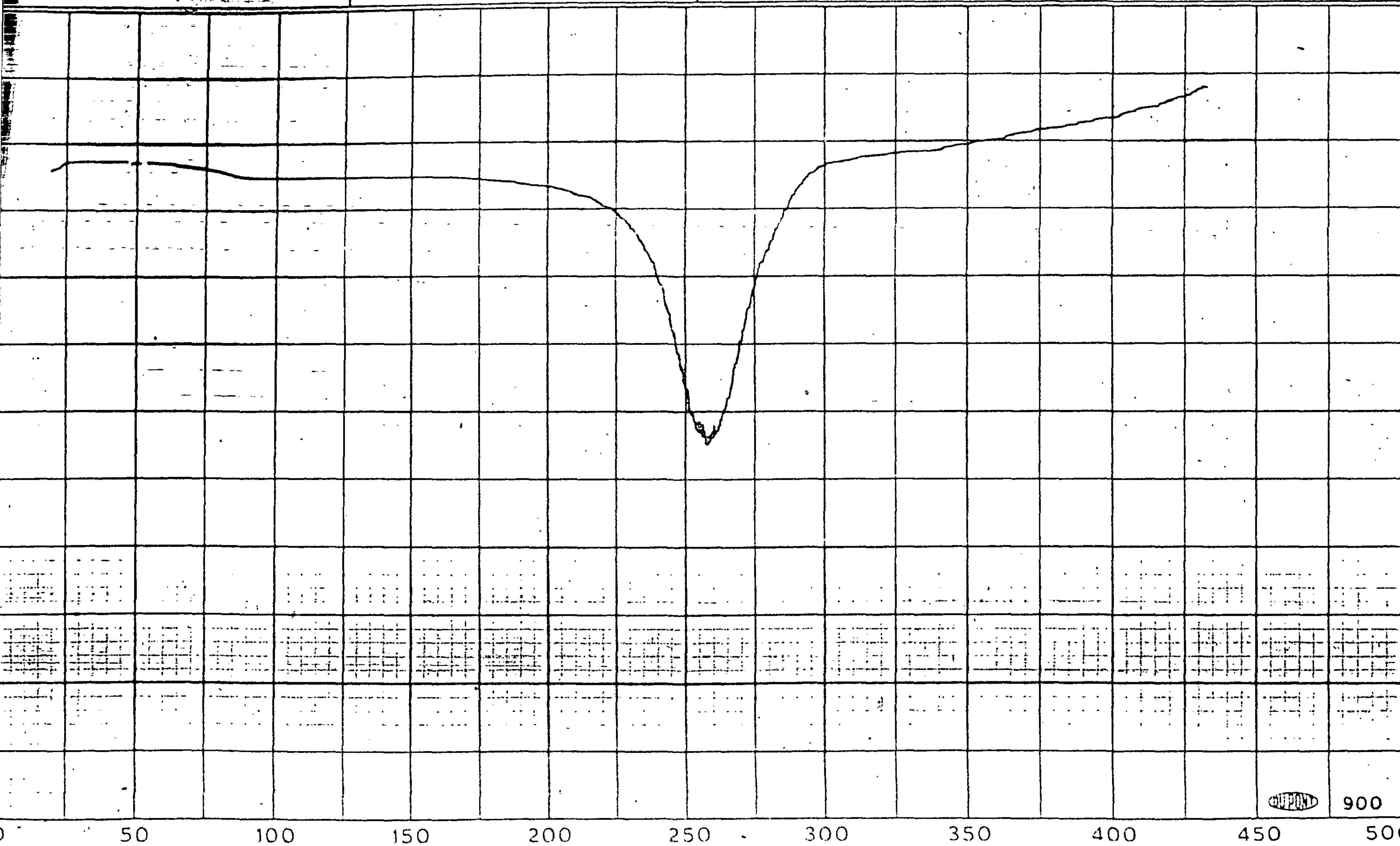


13

SAMPLE: Pts Plato Lot No 553L Part No. 805039 ORIGIN:	SIZE 2mm in depth REF. glass beads PROGRAM MODE heat.. RATE 7.2 °C/MIN, START 25 °C	ATM. SCALE SETTING: T 50 °C/IN.; ΔT 1.0 °C/IN.	RUN NO. #16 DATE 7.21.80 OPERATOR Staff

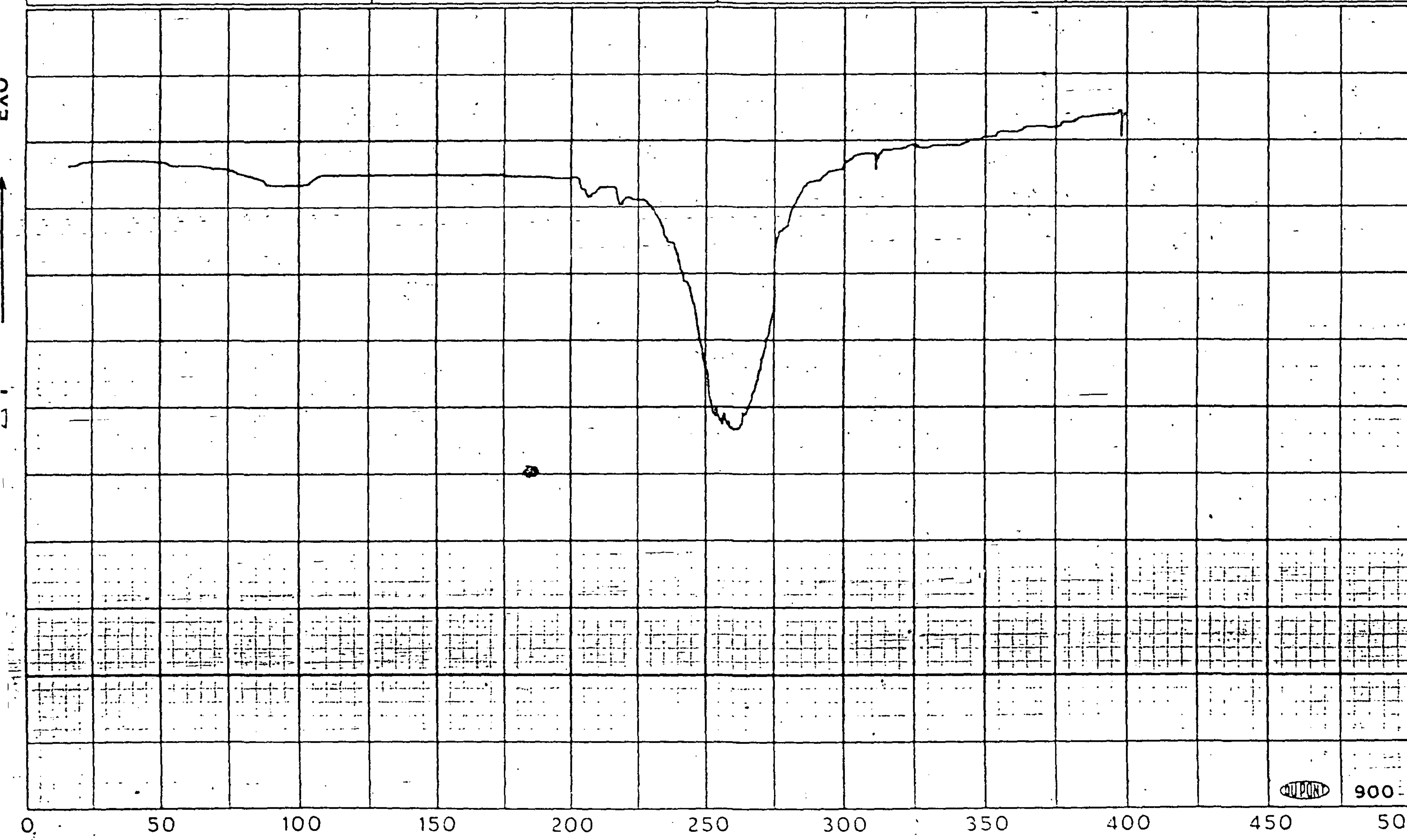


SAMPLE: Pos-[illegible] Lot No [illegible] Changed [illegible] 10mm/5g [illegible] ORIGIN:	SIZE 2 mm deep REF. glass beads PROGRAM MODE heat RATE ___ °C/MIN, START 25°C	ATM. ___ T: SCALE SETTING 50 °C/IN. ΔT: ___ °C/IN.	RUN NO. 17 DATE 7-6-22-8 OPERATOR Steff



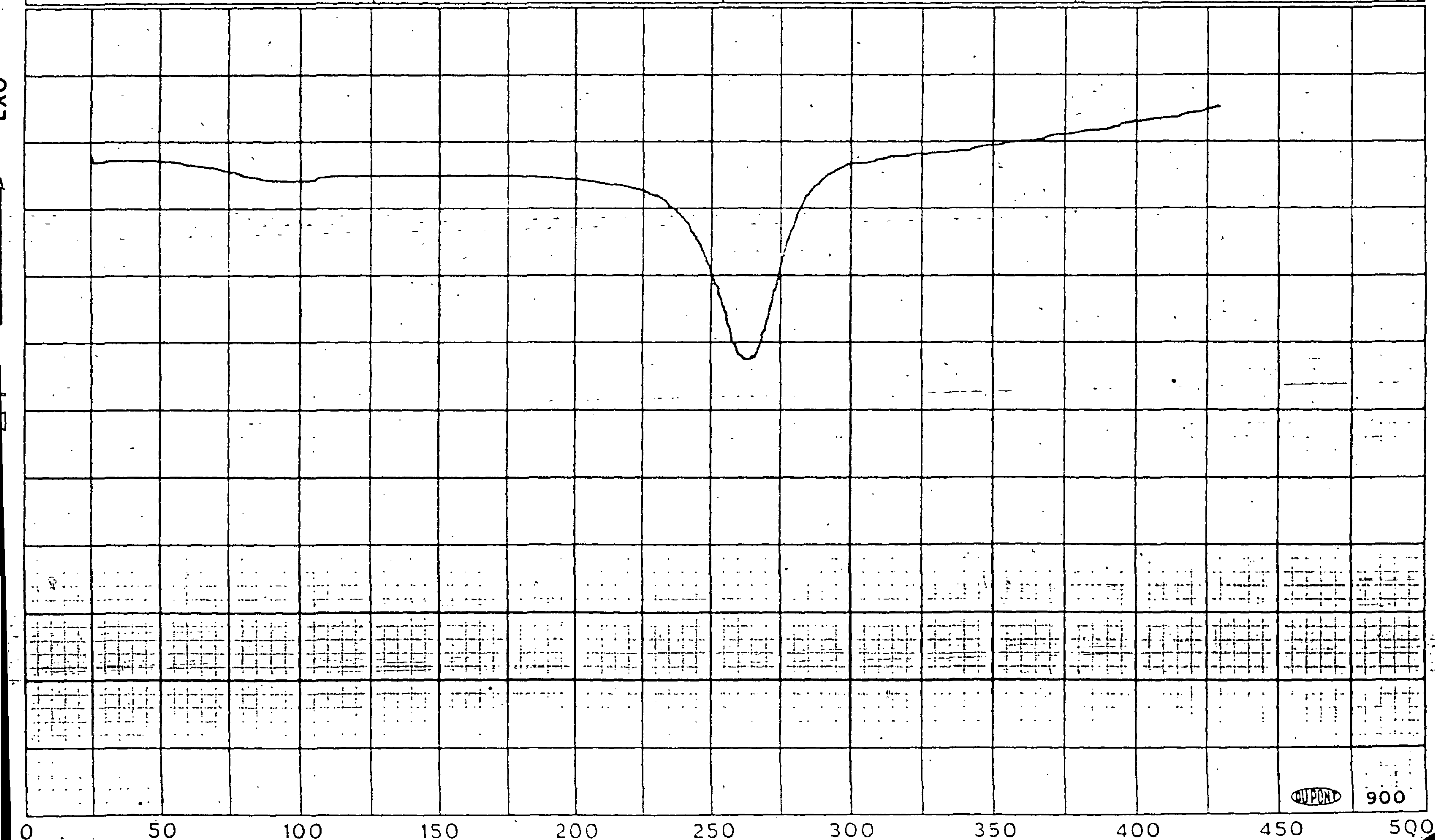
15

SAMPLE: Pos Plate Lot No 5532 Part No 865639 Charged at 20 ma/sq in **ORIGIN:**	**SIZE** 2mm deep **REF.** glass beads **PROGRAM MODE** Heat **RATE** ___ °C/MIN, **START** 25 °C	**ATM.** ___ **SCALE SETTING** T: 50 °C/IN. ΔT: 1.0 °C/IN.	**RUN NO.** 18 **DATE** 7.24.80 **OPERATOR** Staff



1K

SAMPLE: Pos Plate Lot NO 553L Part NO 905059 charged at 30 m/sq in ORIGIN:	SIZE 2 mm deep REF. glass beads PROGRAM MODE heat RATE 7.0 °C/MIN. START 25 °C	ATM. SCALE SETTING: T 50 °C/IN.; ΔT 1.0 °C/IN.	RUN NO. 19 DATE 7.28.80 OPERATOR Staff



DU PONT INSTRUMENT PRODUCTS DIVISION

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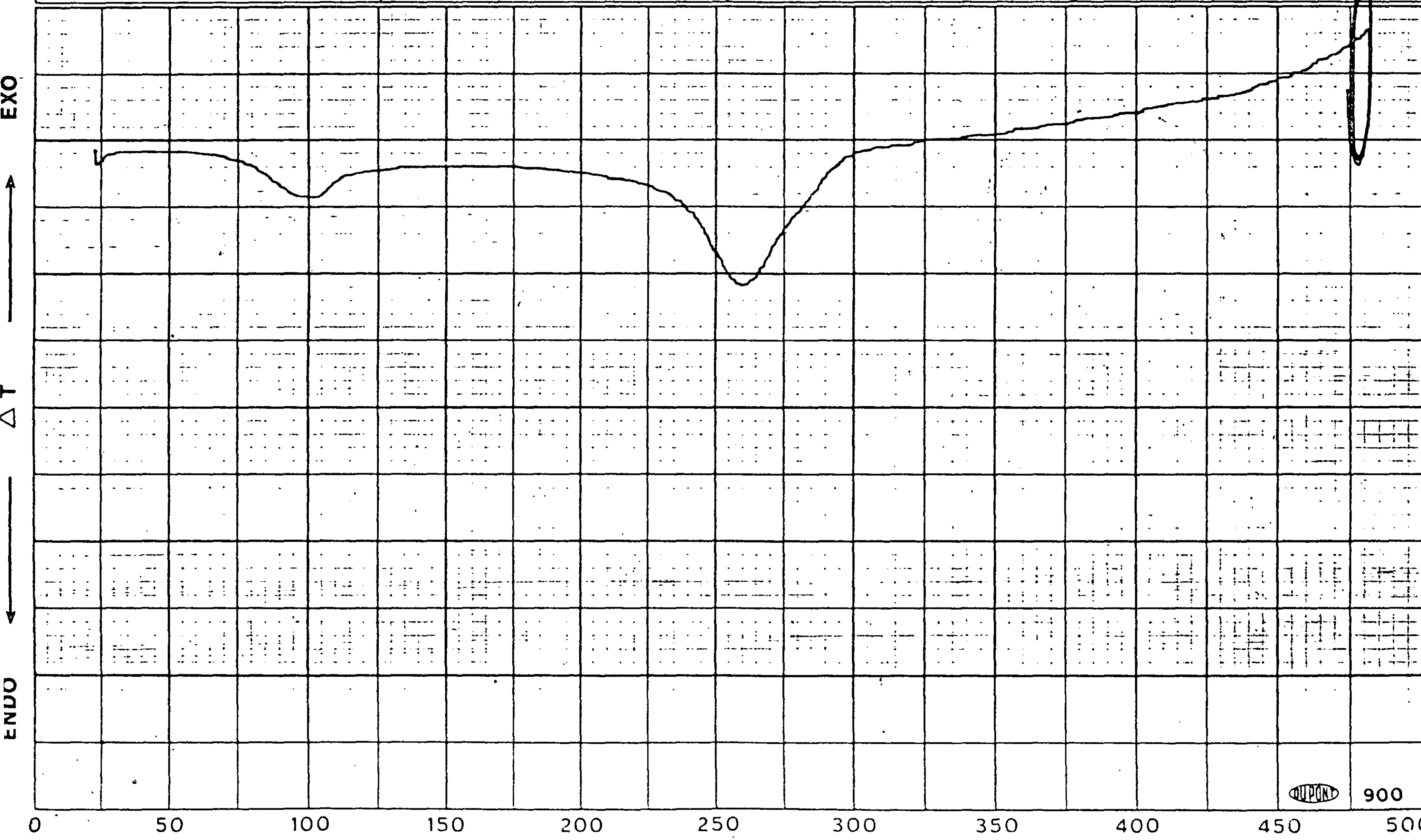
SAMPLE: Pos Plate Lot NO 553L Part No. 305039 Charged at 40ma/sq in ORIGIN:	SIZE 2 mm deep REF. glass beads PROGRAM MODE heat RATE -7 °C/MIN, START 25°C	ATM. SCALE SETTING: T 50 °C/IN. ; ΔT 1.0 °C/IN.	RUN NO. 20 DATE 7.30.80 OPERATOR JTeff



EXPERIMENTAL PART II

ATOMIC ABSORPTION SPECTROSCOPY

Data for Graph I

Analysis of cell GE 12AM S/N01 plates #3, #9, #13

Calibration curve for Ni

Table Ia

PPM	A.A. Reading	%Abs
2	.0234	5.3
4	.044	9.7
6	.0644	13.8
8	.0842	17.6
10	.1011	22.4

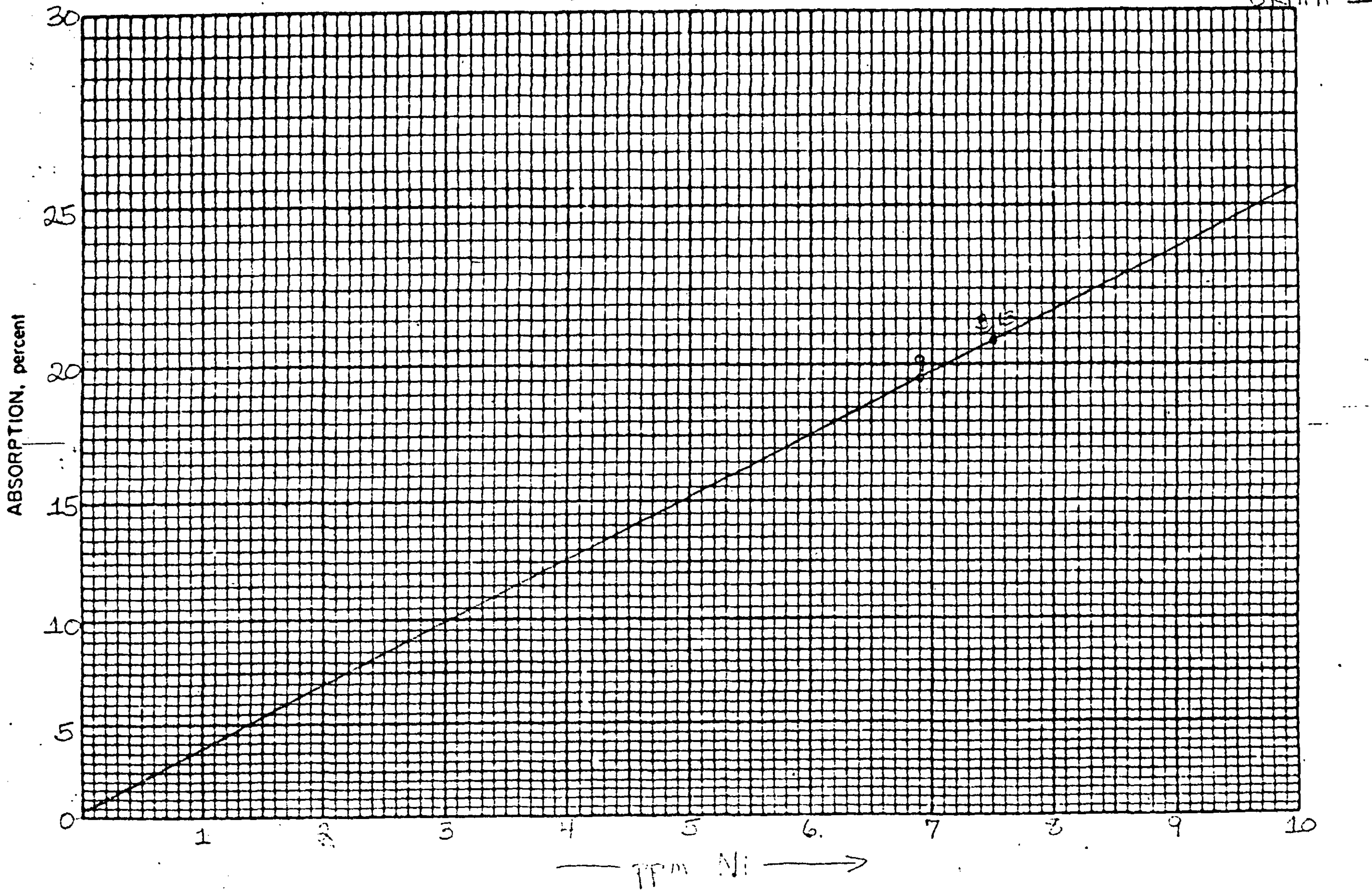
Unknown sample analyses

Table Ib

Sample No.	Dilution Factor	A.A. Reading	%Abs	PPM	PPM Original Solution
GE 12AM S/N01 #3	X 10	.079	16.7	7.5	75.0
GE 12AM S/N01 #9	X 10	.0734	15.6	6.9	69.0
GE 12AM S/N01 #13	X 10	.0802	16.9	7.5	70.0

ANALYSES OF SAMPLES GE 12 AM SN 01 #3, #9, AND #15

GRAPH 1



Data for Graph II

Ni analyses of cell GE 02 plates #2, #8, #12

Calibration curve for Ni

Table IIa

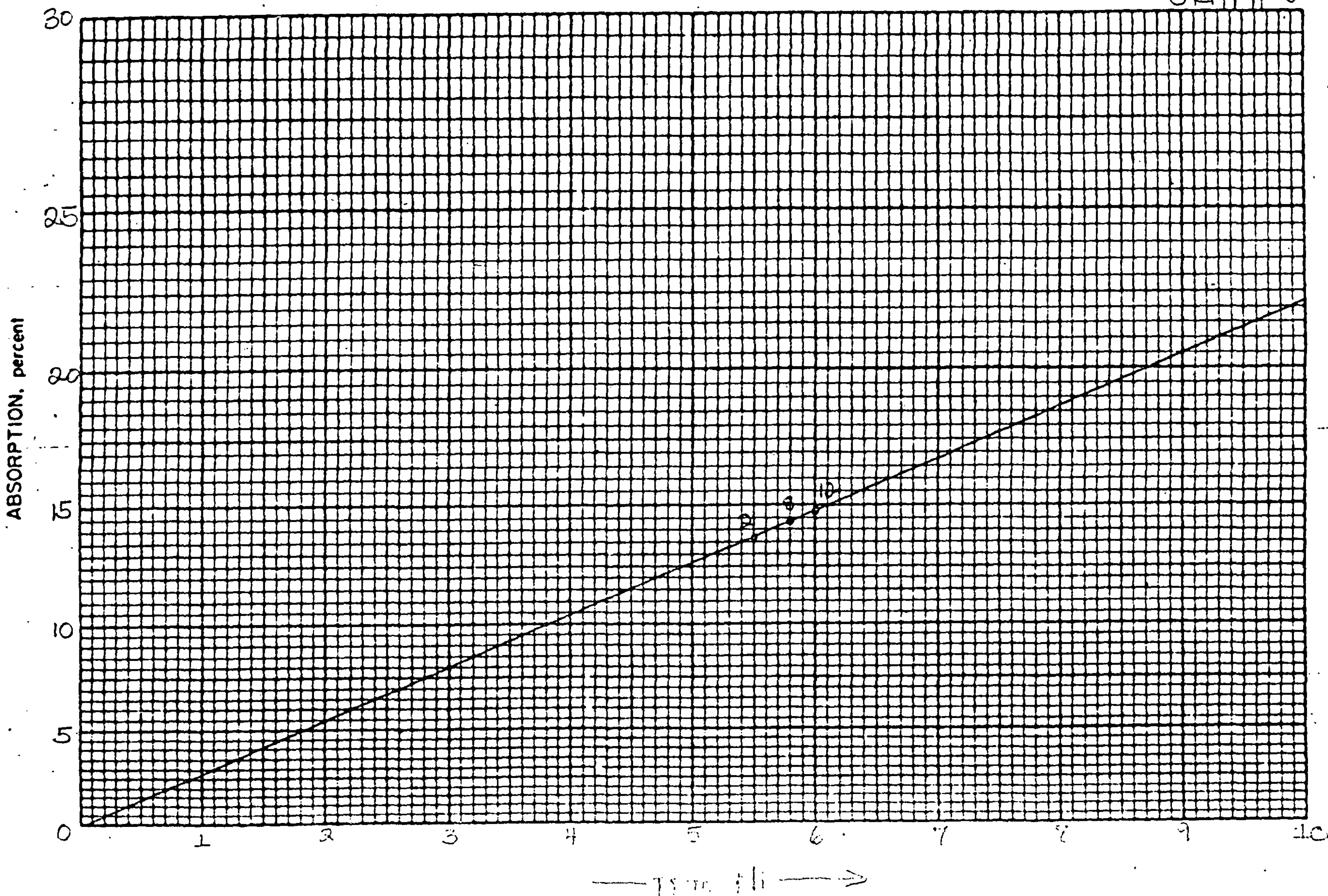
PPM	A.A. Reading	%Abs
2	.030	8.0
4	.056	12.2
6	.082	17.3
8	.109	22.2
10	.128	25.5

Unknown sample analyses

Sample No.	Dilution Factor	A.A. Reading	%Abs	PPM	PPM Original Solution
GE 02 #2	250	.076	16.1	5.51	1380
GE 02 #8	250	.081	17.0	5.80	1480
GE 10 #12	250	.084	17.6	6.00	1500

ANALYSES OF SAMPLES GE O2 POSITIVE, #2, #8, AND #12

GRAPH 2



Data for Graph III

Ni analyses of cell GE 02 Positive plate #12

Calibration curve for Ni

Table IIIa

PPM	A.A. Reading	%Abs
2	.0326	7.2
4	.576	12.4
6	.0834	17.5
8	.1088	22.3
10	.130	25.9

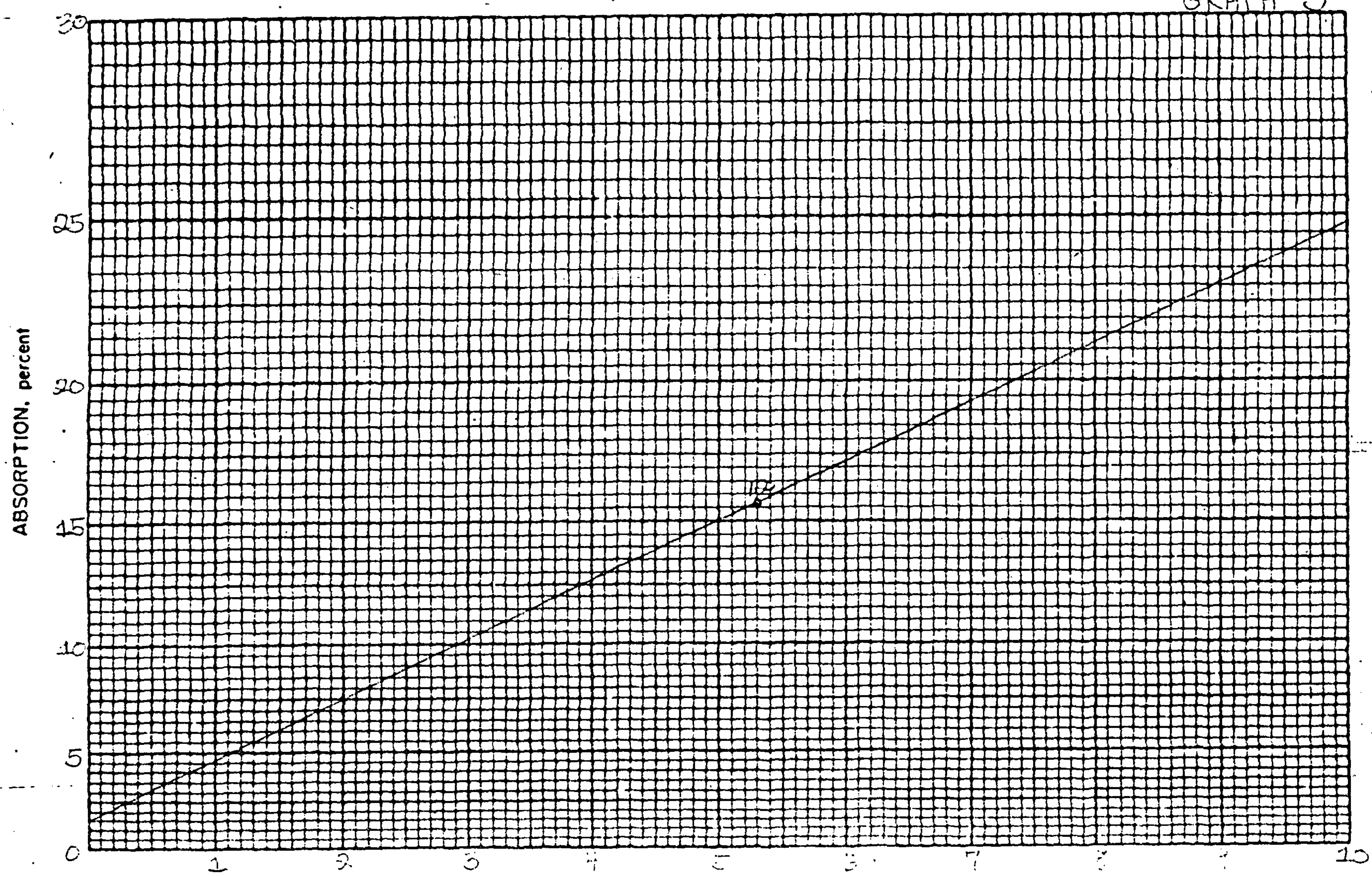
Unknown sample analysis

Table IIIb

Sample No.	Dilution Factor	A.A. Reading	%Abs	PPM	PPM Original Solution
GE 02 Positive #12	.250	.0745	15.8	5.30	1320

ANALYSES OF SAMPLE GE 02 POSITIVE #12

GRAPH 3



Data for Graph IV

Ni analyses of cell GE 02 plates #3, #9, #13

Calibration curve for Ni

Table IVa

PPM	A.A. Reading	%Abs
2	.021	4.7
4	.0398	7.8
6	.0622	13.3
8	.0802	15.9
10	.0920	19.2

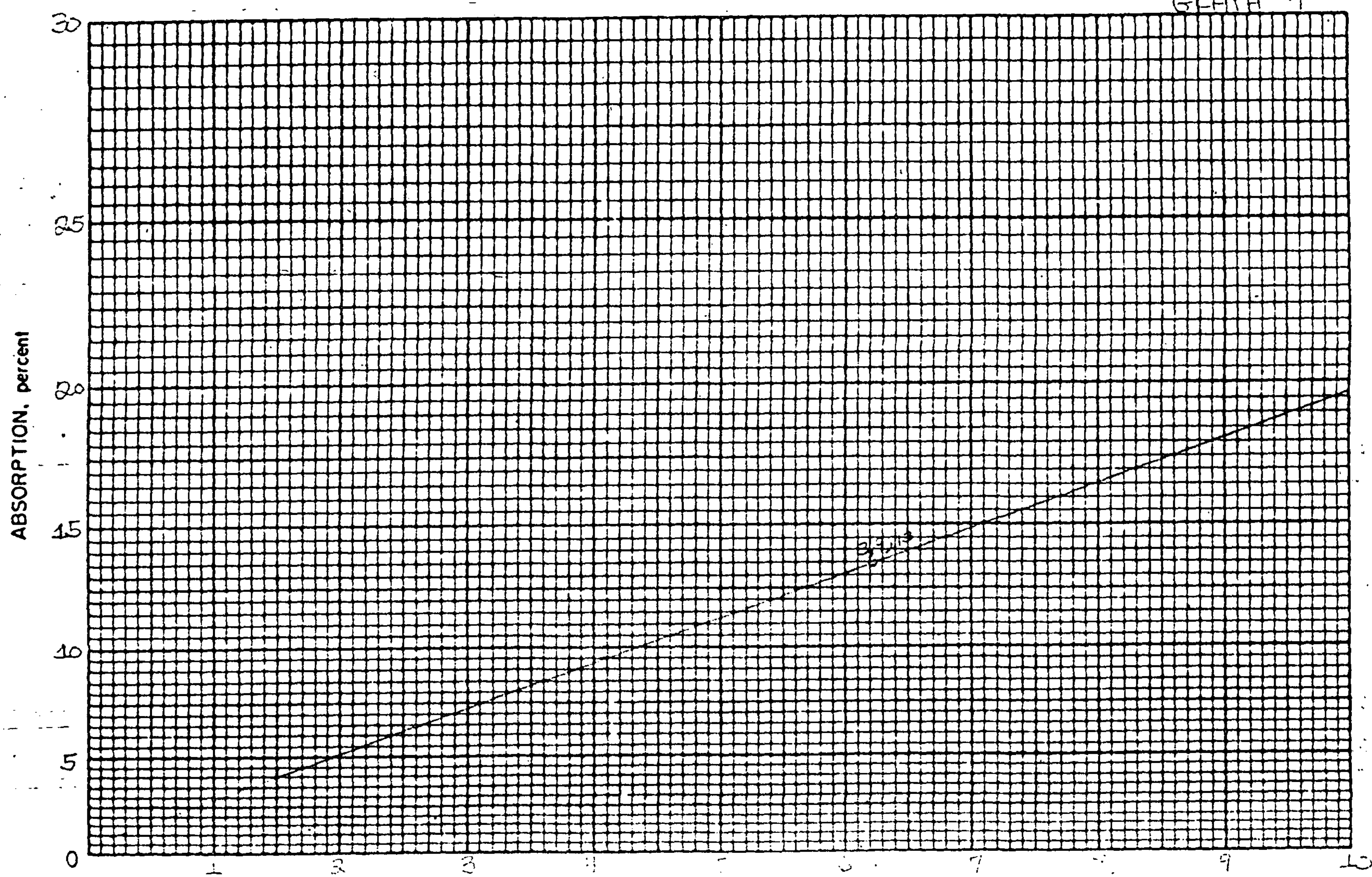
Unknown sample analyses

Table IVb

Sample No.	Dilution Factor	A.A. Reading	%Abs	PPM	PPM Original Solution
GEO AMP #3	250	.060	12.9	6.23	1557.0
GEO AMP #9	250	.060	12.9	6.23	1557.0
GEO AMP #13	250	.060	12.9	6.23	1557.0

ANALYSIS OF SAMPLES GE O2 AM. #5, #9, AND #15

GRAPH 4



Data for Graph V

Ni analyses of cell 12 AM SNO_2 plates #3, #9, #13

Calibration curve for Ni

Table Va

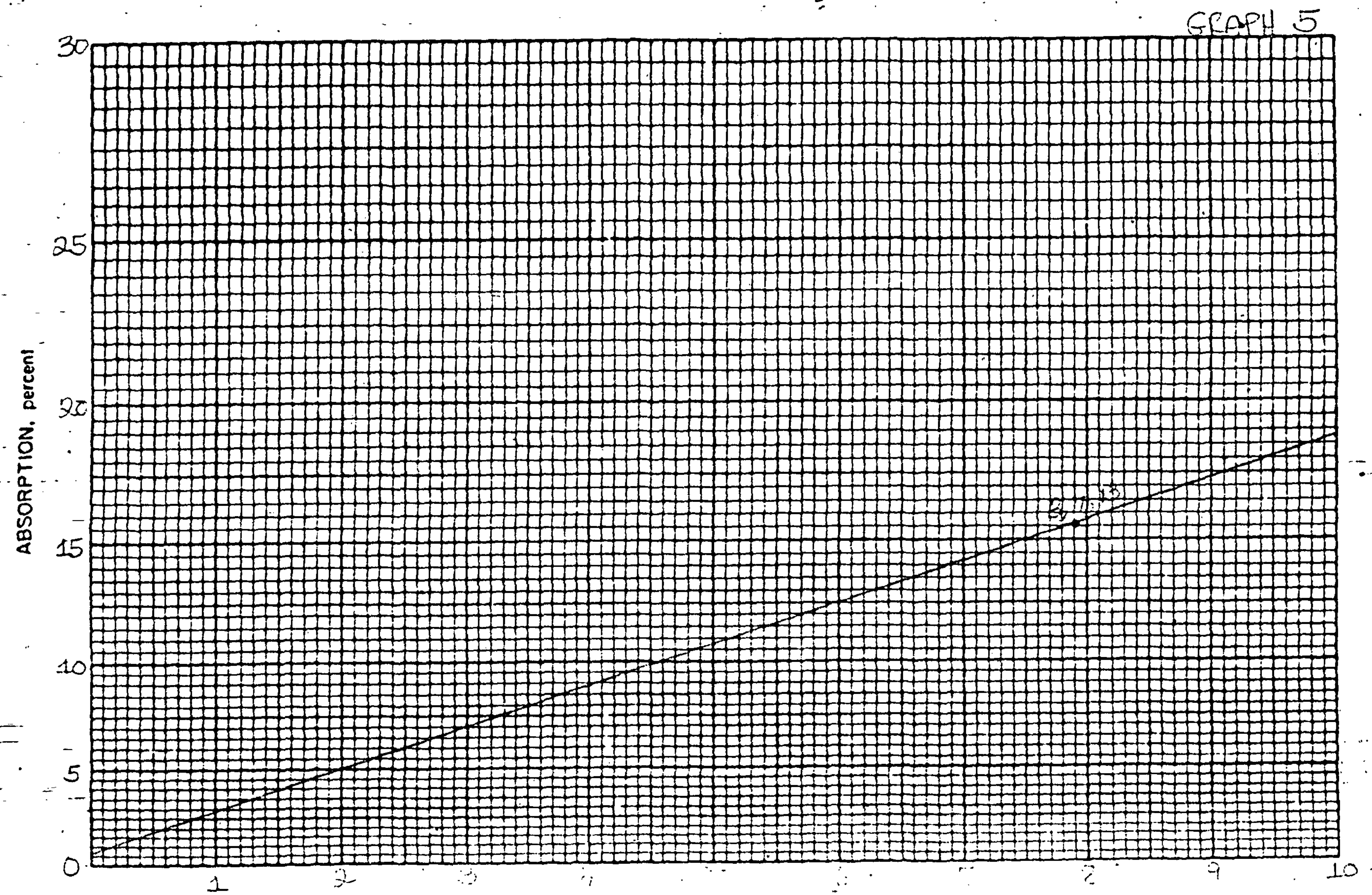
PPM	A.A. Reading	%Abs
2	.022	5.0
4	.040	9.1
6	.060	13.0
8	.079	16.7
10	.095	19.3

Unknown sample analyses

Table Vb

Sample No.	Dilution Factor	A.A. Reading	%Abs	PPM	PPM Original Solution
12AM SNO_2 #3	10	.077	16.3	7.9	79.0
" #9	10	.080	16.5	7.9	79.0
" #13	10	.078	16.4	7.9	79.0

ANALYSES OF SAMPLES 12 AM SN 02 #5, #9, AND #13



Data for Graph VI

Ni analyses of cell GE 056 plates #3, #9, #13

Calibration curve for Ni

Table VIa

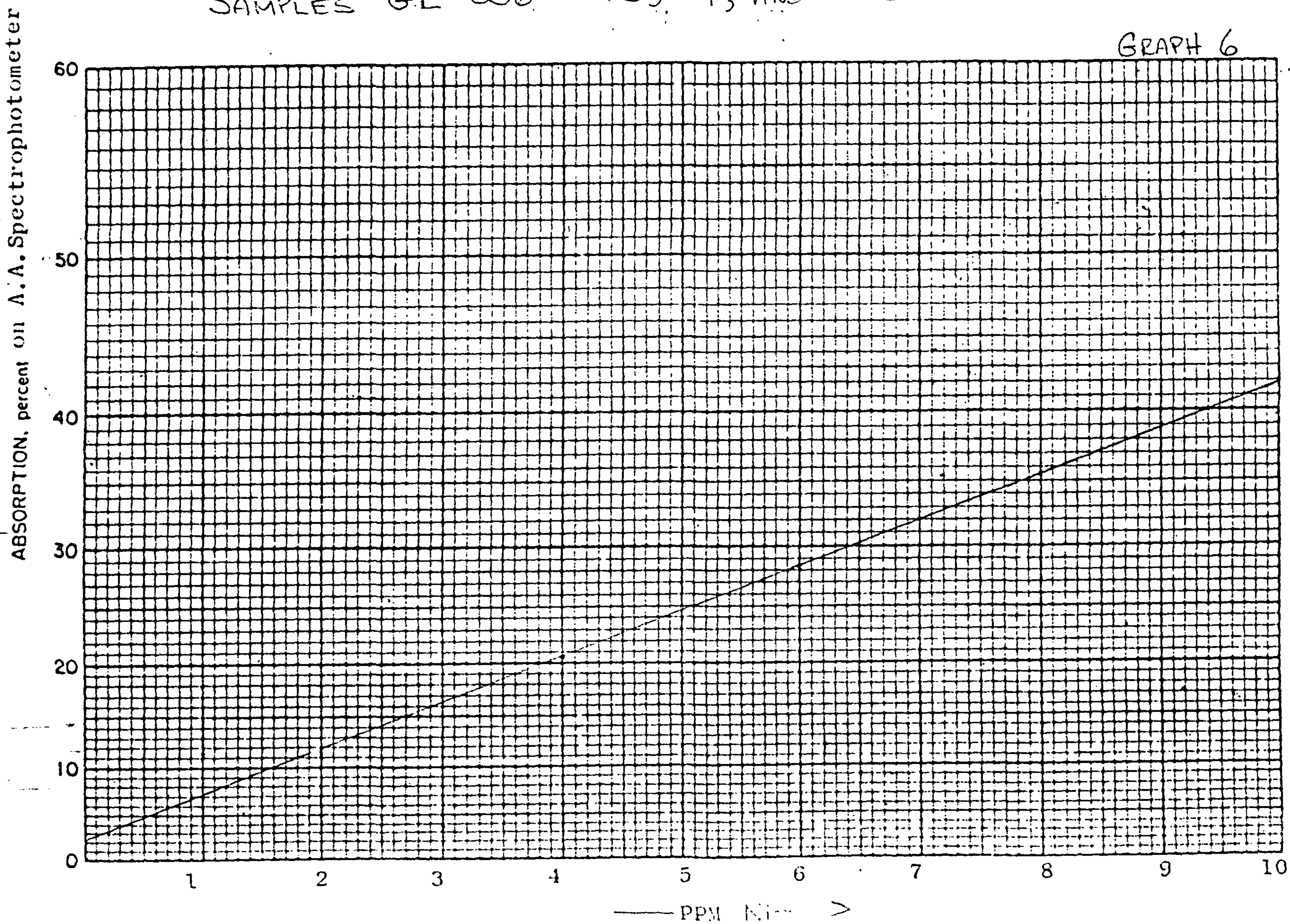
PPM	A.A. Reading	%Abs
2	.053	11.5
4	.100	20.6
6	.144	28.2
8	.190	35.4
10	.220	39.7

Unknown sample analyses

Table VIb

Sample No.	Dilution Factor	A.A. Reading	%Abs	PPM	PPM Original Solution
GE 056 #3	10	.195	36.2	8.30	83.0
" #9	10	.198	36.6	8.42	84.2
" #13	10	.205	37.6	8.72	87.2

SAMPLES GE 056 #3, #9, AND #13



Data for Graph VII

Ni analyses of cell Ge 056 plates #3, #9, #13, AM Extract

Calibration curve for Ni

Table VIIa

PPM	A.A. Reading	%Abs
2	.0498	10.9
4	.0956	19.8
6	.141	27.8
8	.182	34.3
10	.215	39.1

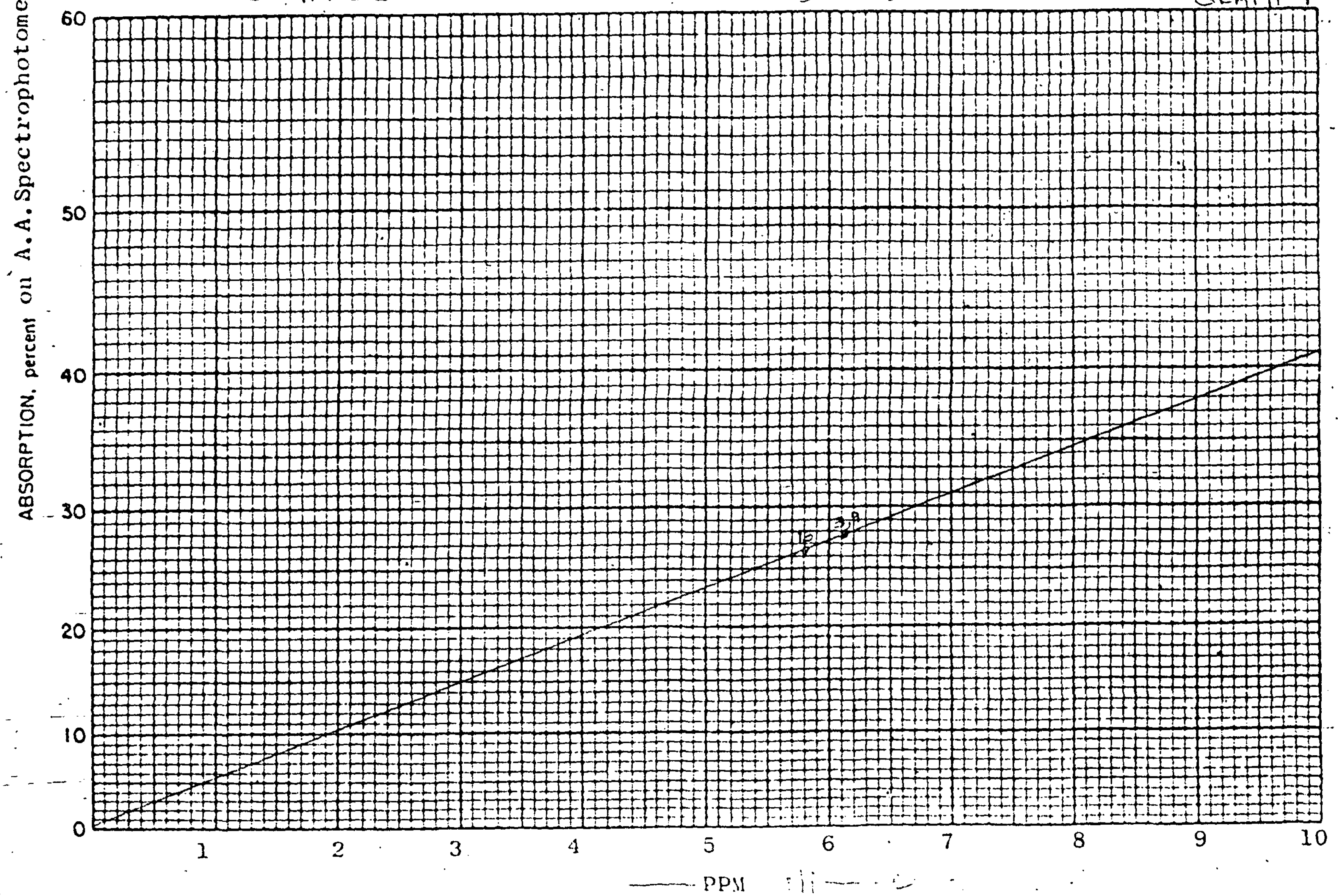
Unknown sample analyses

Table VIIb

Sample No.	Dilution Factor	A.A. Reading	%Abs	PPM	PPM Original Solution
GE 056 #3	250	.142	27.9	6.13	1532.0
" #9	250	.141	27.8	6.13	1532.0
" #13	250	.136	26.9	5.8	1450.0

SAMPLES GE 056 AM #3, #9, and #13

GRAPH 7



Data for Graph VIII

Ni analyses of cell S 01 plates #2, #3, #12

Calibration curve for Ni

Table VIIIa

PPM	A.A. Reading	%Abs
2	.031	6.8
4	.058	12.5
6	.085	17.7
8	.112	22.8
10	.136	26.9

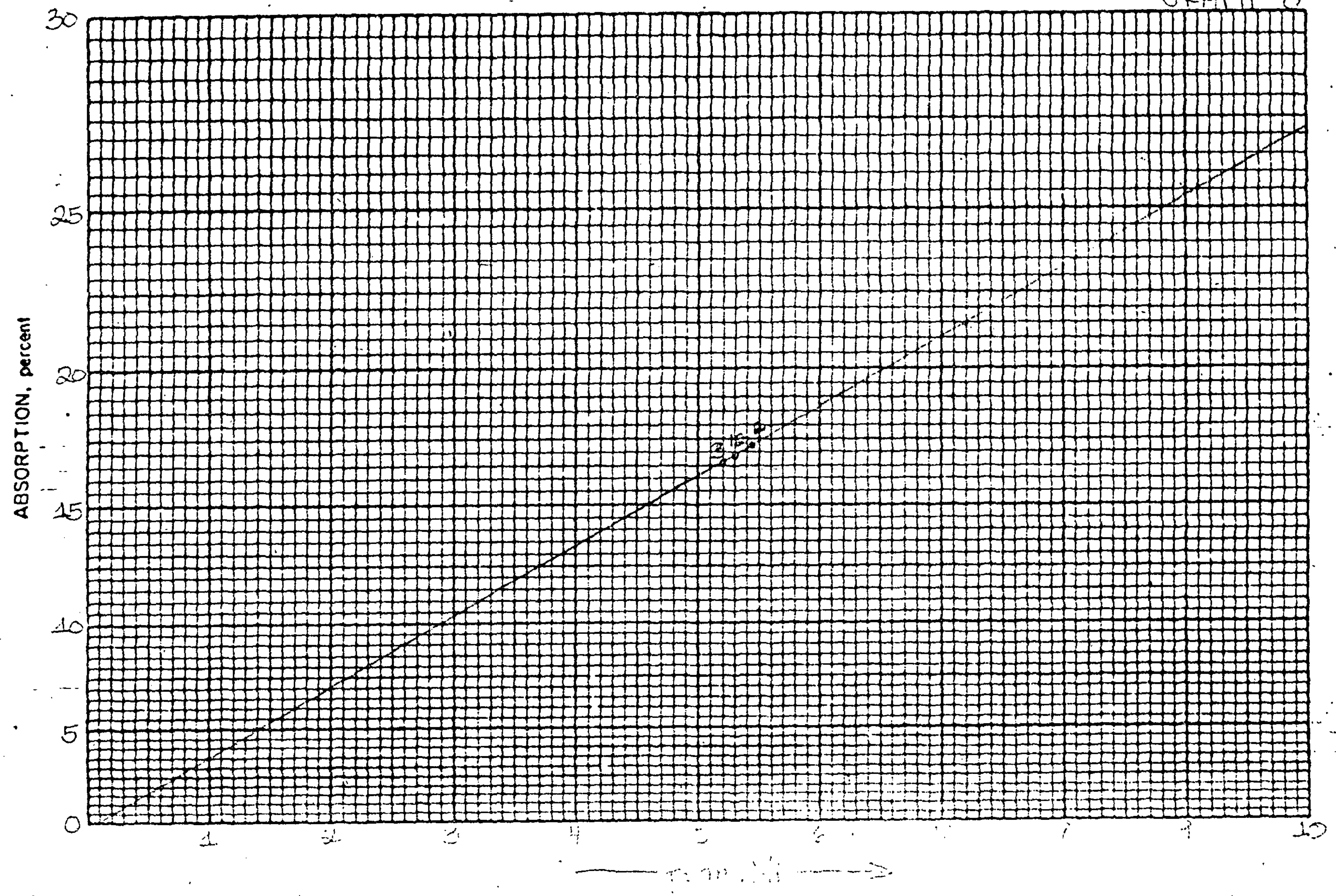
Unknown sample analyses

Table VIIIb

Sample No.	Dilution Factor	A.A. Reading	%Abs	PPM	PPM Original Solution
SN 01 #2	250	.077	16.2	5.43	1357.0
SN 01 #3	250	.073	15.5	5.2	1300.0
SN 01 #12	250	.075	15.8	5.3	1325.0

ANALYSES OF SAMPLES SN 01 POSITIVE #2, #6 AND #12

GRAPH 8



Data for Graph IX

Calibration curve for Ni

Table IXa

PPM	A.A. Reading	%Abs
2	.023	5.1
4	.045	9.8
6	.064	13.7
8	.083	17.3
10	.099	20.3

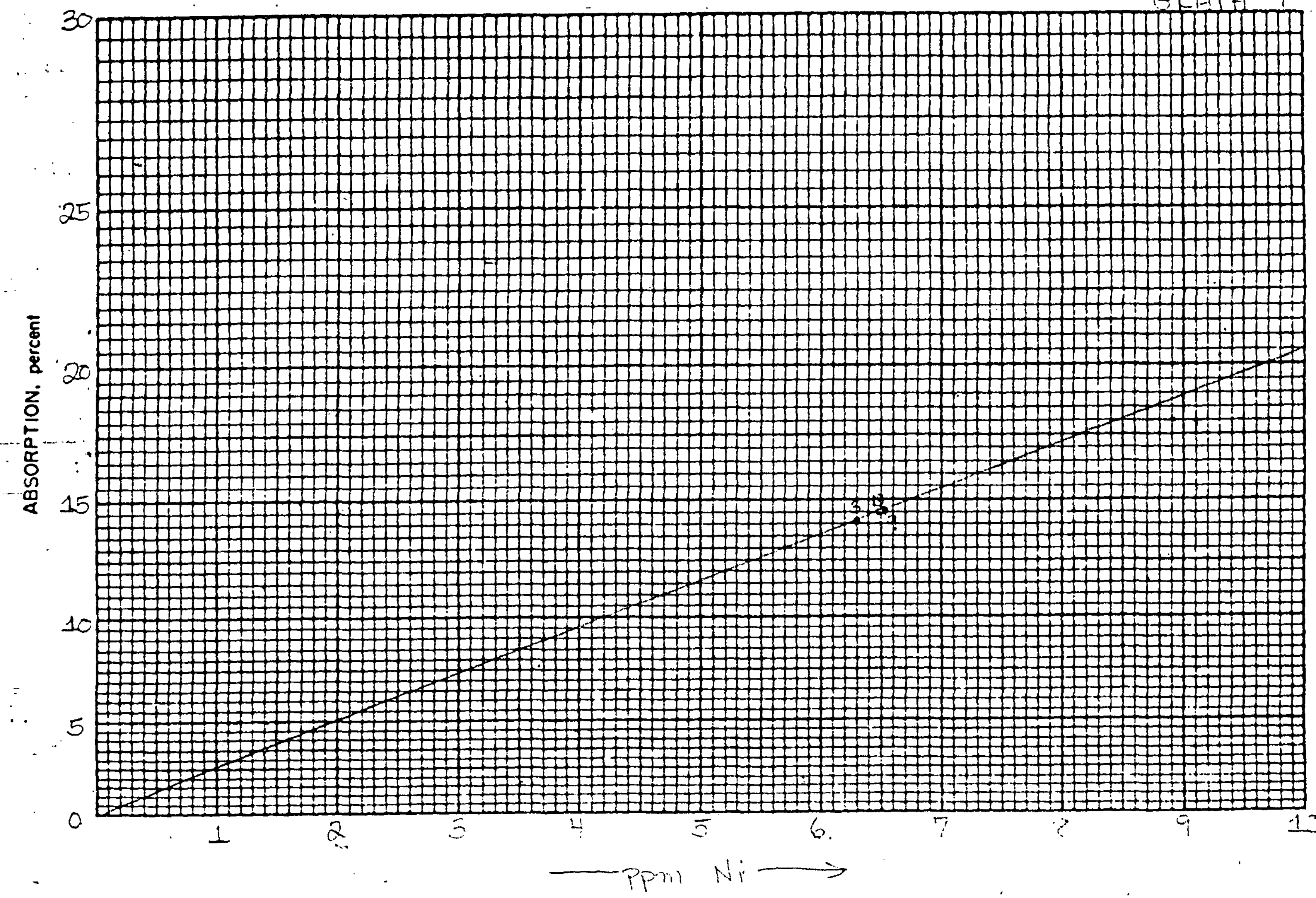
Unknown sample analyses

Table IXb

Sample No.	Dilution Factor	A.A. Reading	%Abs	PPM	PPM Original Solution
GE 02 S/N 01					
" #3	250	.066	14.0	6.3	1575.0
#9	250	.0664	14.2	6.52	1630.0
#13	250	.067	14.4	6.49	1622.5

ANALYSES OF GE 02 #3, #9, AND #15

GRAPH 9



Data for Graph X

Cd analyses of cell GE 12 AM SN 01
Negative plates #3, #9, #13
Positive plates #2, #8, #12

Calibration curve for Cd

Table Xa

PPM	A.A. Reading	%Abs
1	.065	11.5
2	.123	24.6
3	.174	33.0
4	.224	40.3
5	.267	46.0

Unknown sample analyses

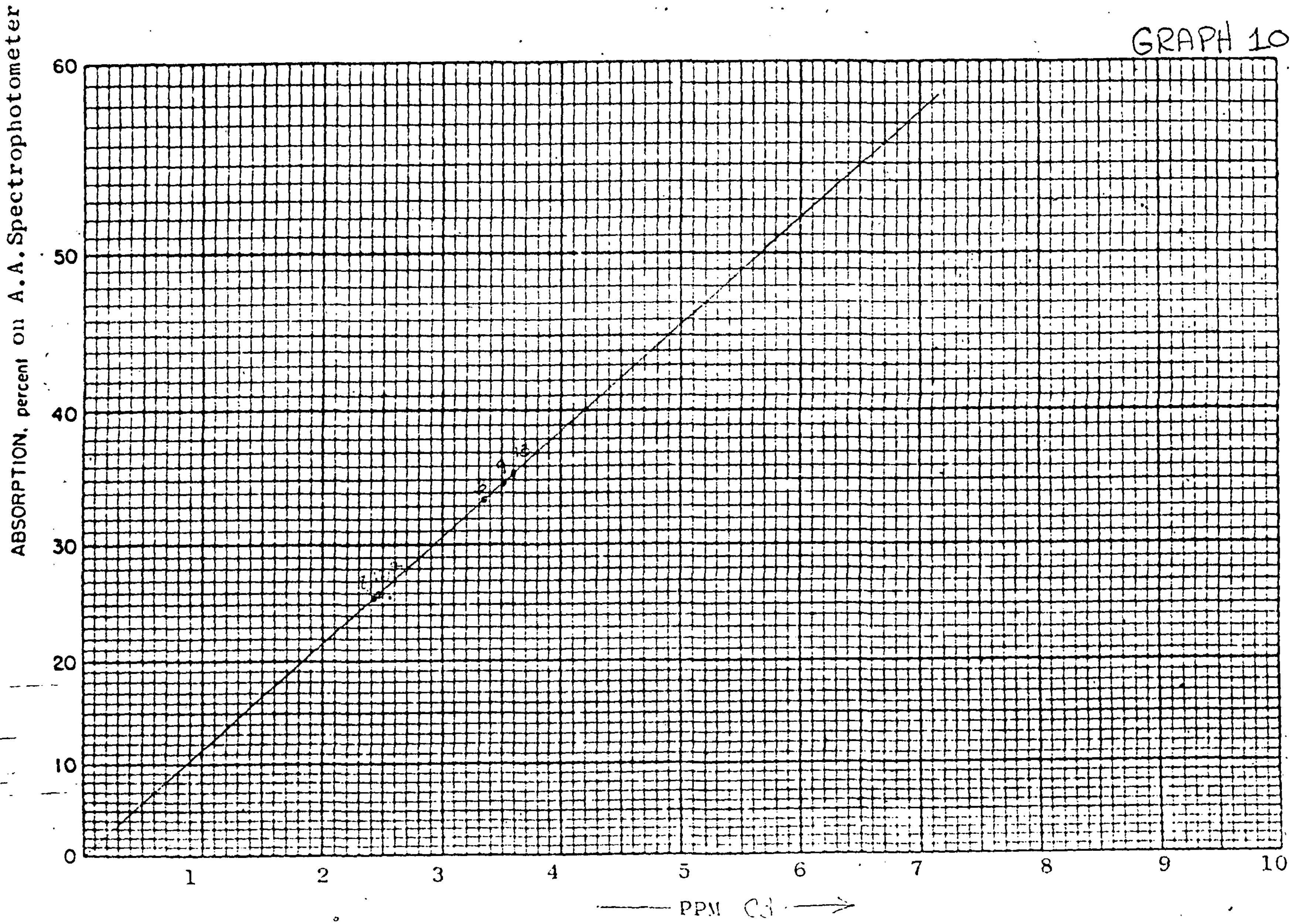
Table Xb

Sample No.	Dilution Factor	A.A. Reading	%Abs	PPM	PPM Original Solution
GE 12AM SN 01	250	.189	35.3	3.35	837
"	250	.199	36.8	3.51	877
"	250	.202	37.2	3.60	900
"	50	.146	28.6	2.49	248
"	50	.144	28.2	2.41	241
"	50	.145	28.4	2.42	242

ANALYSES OF SAMPLES GE 12AM SN 01

NEGATIVE #5, #9 + #13
POSITIVE #2, #8 + #12

GRAPH 10



Data for Graph XI

A run of known Cd solutions was made to make sure of the reproducibility of standard calibration.

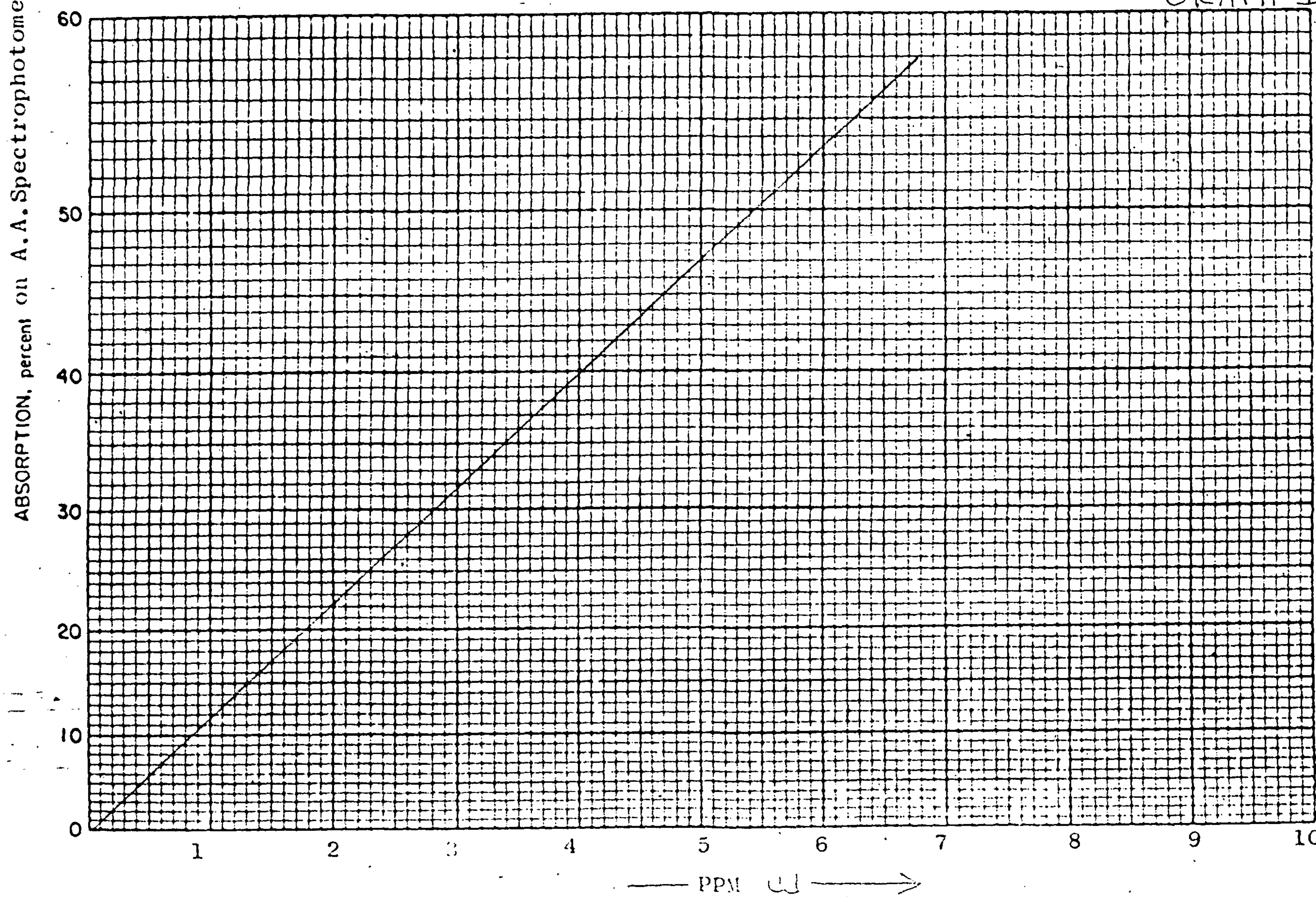
Table XIa

PPM	A.A. Digital Readout	%Abs
1	.0658	14.1
2	.1242	24.9
3	.1766	33.4
4	.255	40.5
5	.2672	46.0

The calibration curve is reproducible.

A CALIBRATION CURVE OF STANDARD Cd SOLUTIONS TO VERIFY REPRODUCIBILITY OF CURVE

GRAPH 11



Data for Graph XII

Calibration curve for Cd

Table XIIa

PPM	A.A. Reading	%Abs
1	.069	14.7
2	.1276	25.5
3	.2278	33.5
4	.2714	40.8
5		46.5

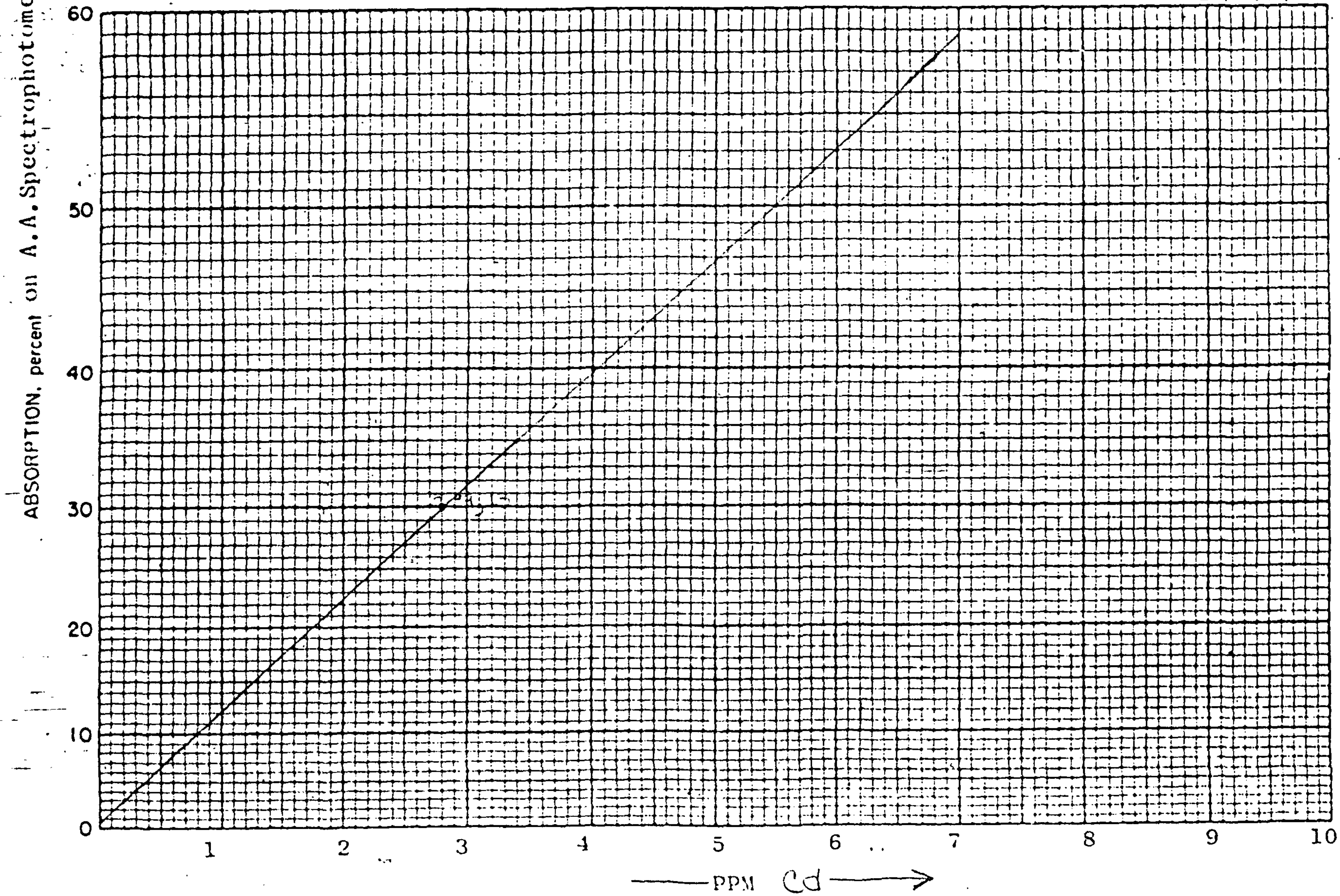
Unknown sample analyses

Table XIIb

Sample No.	Dilution Factor	A.A. Reading	%Abs	PPM	PPM Original Solution
12AM GE 02 #3	250	.1676	32.0	2.8	700
" #9	250	.1744	33.1	2.9	725
" #13	250	.1756	33.3	2.93	732.5

GRAPH 12

SAMPLES 12 AM. SNO_2 GE O2 #3, #9, AND #13



Data for Graph XIII

Cd analyses of cell GE 02 plates #3, #9, #13

Calibration curve for Cd

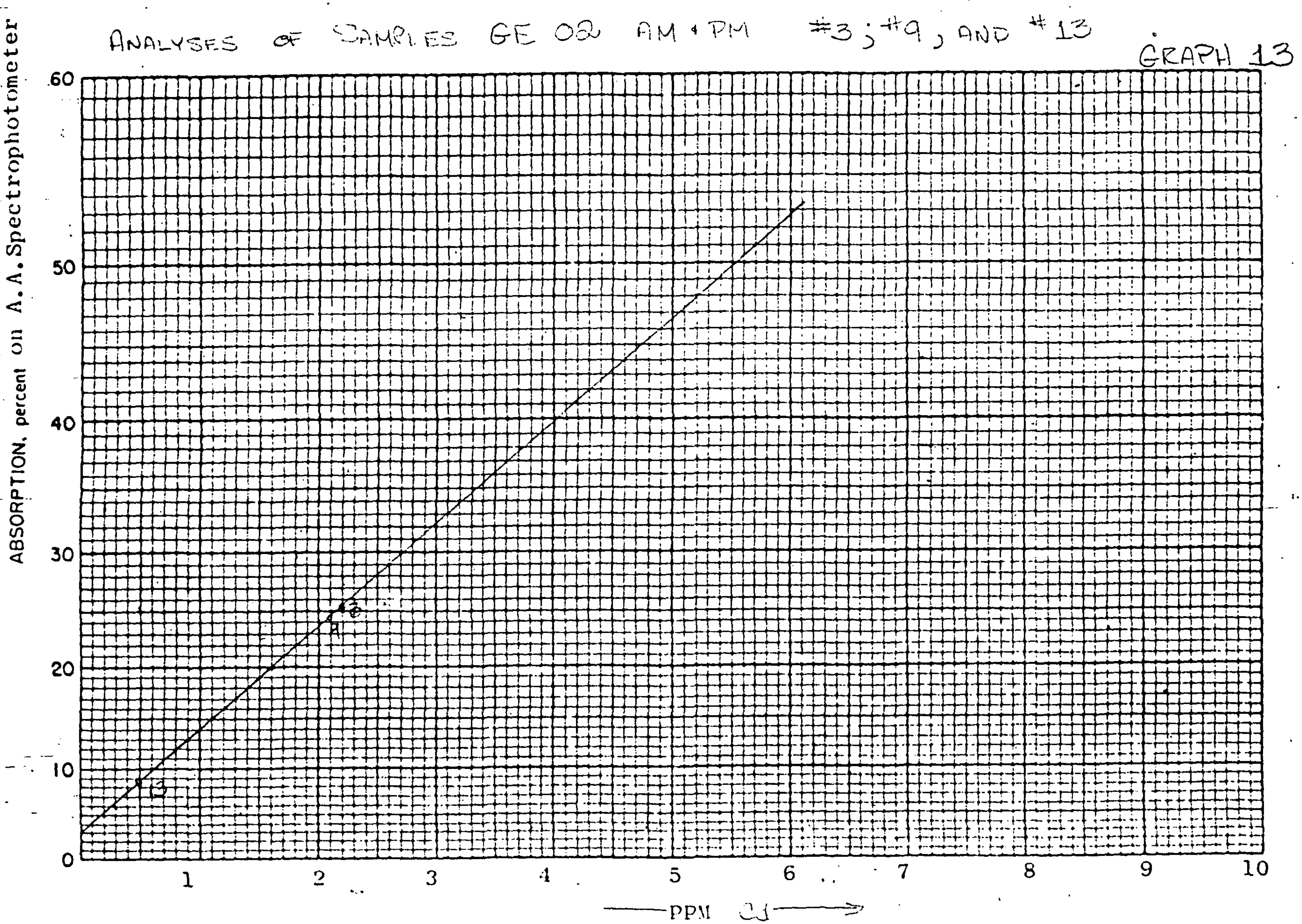
Table XIIIa

PPM	A.A. Reading	%Abs
1	.0646	13.8
2	.1256	25.2
3	.1764	33.4
4	.2244	40.4
5	.2738	45.4

Unknown sample analyses

Table XIIIb

Sample No.	Dilution Factor	A.A. Reading	%Abs	PPM	PPM Original Solution
GE 02 #3	0	.1356	26.8	2.20	2.20
" #9	0	.123	24.7	2.10	
" #13	0	.0322	7.1	0.5	0.50



ANALYSES OF SAMPLES GE 02 AM + PM #3; #9, AND #13
GRAPH 13
ABSORPTION, percent on A.A. Spectrophotometer
0
10
20
30
40
50
60
1
2
3
4
5
6
7
8
9
10
PPM Cu
13

Data for Graph XIV

Cd analyses of cell 12 AH SNO_2 GE02 plates #3, #9, #13

Calibration curve for Cd

Table XIVa

PPM	A.A. Reading	%Abs
1	.069	14.7
2	.128	25.5
3	.177	33.5
4	.228	40.8
5	.271	46.5

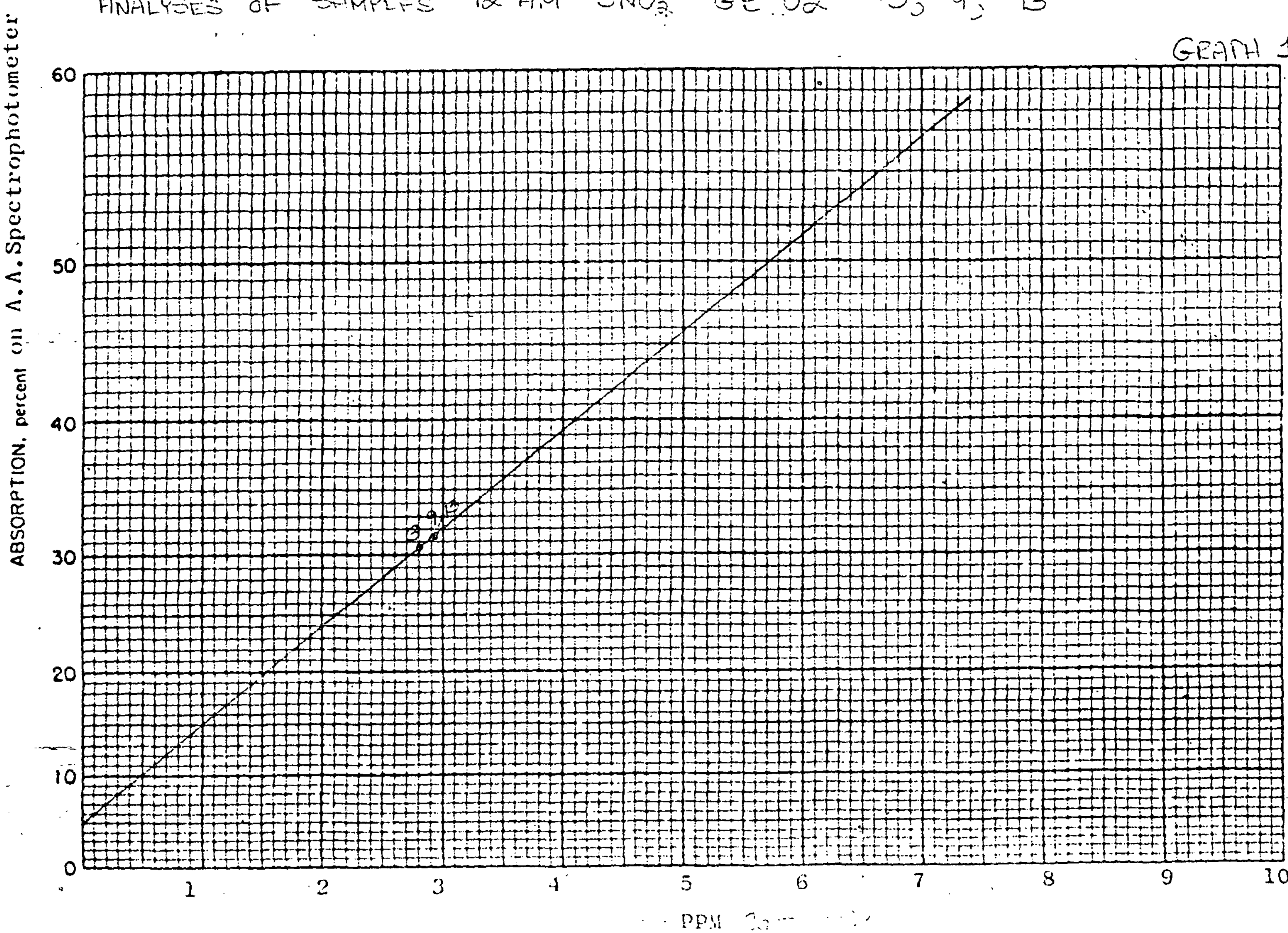
Unknown sample analyses

Table XIVb

Sample No.		Dilution Factor	A.A. Reading	%Abs	PPM	PPM Original Solution
12 AH SNO_2						
GE 02	#3	250	.168	32.0	2.80	700.0
"	#9	250	.174	33.1	2.90	725.0
"	#13	250	.176	33.3	2.93	732.5

ANALYSES OF SAMPLES 12 AM SnO_2 GE 02 #3, #9, #13

GRAPH 14



Data for Graph XV

Cobalt analysis of cell GE 056 plates, #2, #8, #12

Calibration curve for Co

Table XVa

PPM	A.A. Reading	%Abs
1	.020	4.5
2.5	.051	11.1
4	.078	16.5
5	.096	19.9
6	.113	22.9
8	.1465	28.6
10	.173	32.9

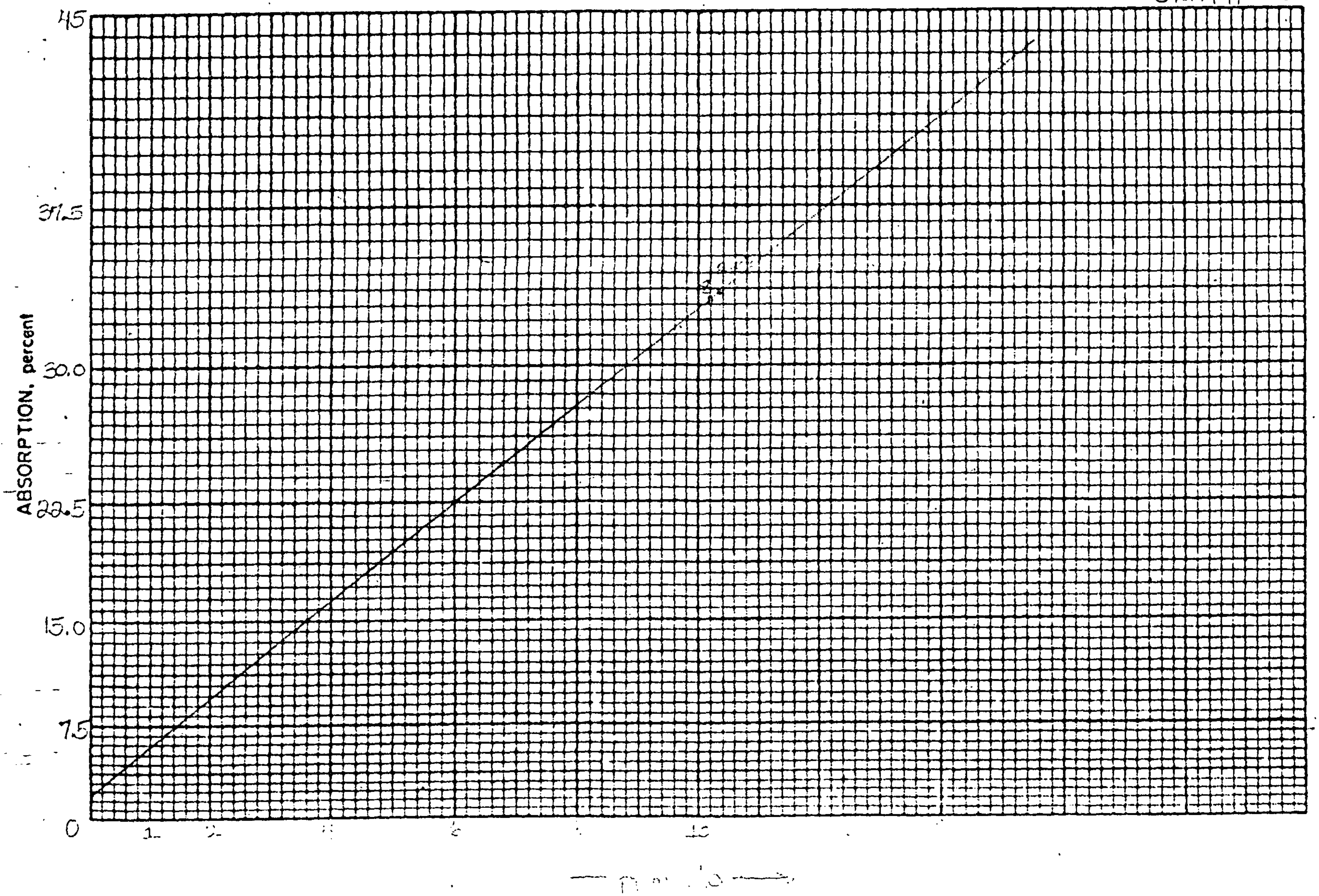
Unknown sample analyses

Table XVb

Sample No.	Dilution Factor	A.A Reading	%Abs	PPM	PPM Orig. Solution
GE 056 #2	5	.183	34.4	10.35	52.0
" #8	5	.179	33.8	10.2	51.0
" #12	5	.181	34.1	10.3	52.0

COLD FINGER EXTRACT GE 056. #2, #8, AND #12

GRAPH 15



Data for Graph XVI

Co analyses of cell GE 02 Positive Plates #2, #8, #12

Calibration curve for Co

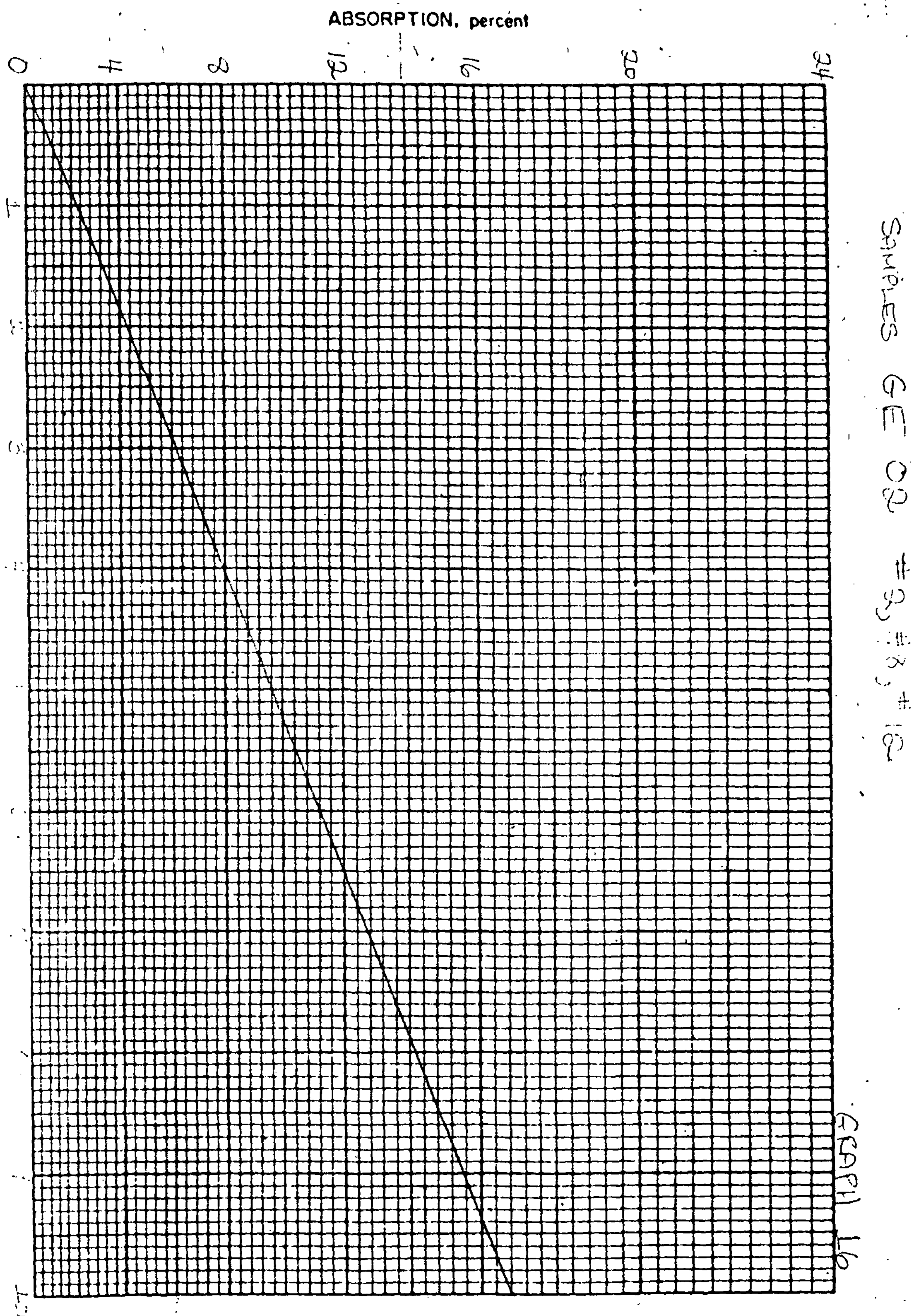
Table XVIa

PPM	A.A. Reading	%Abs
1	.010	2.3
2.5	.023	5.2
4	.036	8.0
5	.043	9.5
6	.051	11.1
8	.067	14.3
10	.081	17

Unknown sample analyses

Table XVIb

Sample No.	Dilution Factor	A.A Reading	%Abs	PPM	PPM Orig. Solution
GE 02 #2	5	.083	17.4	10.15	51.0
" #8	5	.086	17.8	10.35	52.0
" #12	5	.081	17.0	10.00	50.0


ABSORPTION, percent
0
4
8
12
16
20
24
SAMPLES GE 02 #3, #8, #16
GRAPH 16

Data for Graph XVII

Co analyses of cell GE 12 SN/01 plates #2, #8, #12

Calibration curve for Co

Table XVIIa

PPM	A.A. Reading	%Abs
1	.007	1.6
2.5	.019	4.3
4	.030	6.7
5	.037	8.2
6	.044	9.6
8	.058	12.5
10	.070	14.9

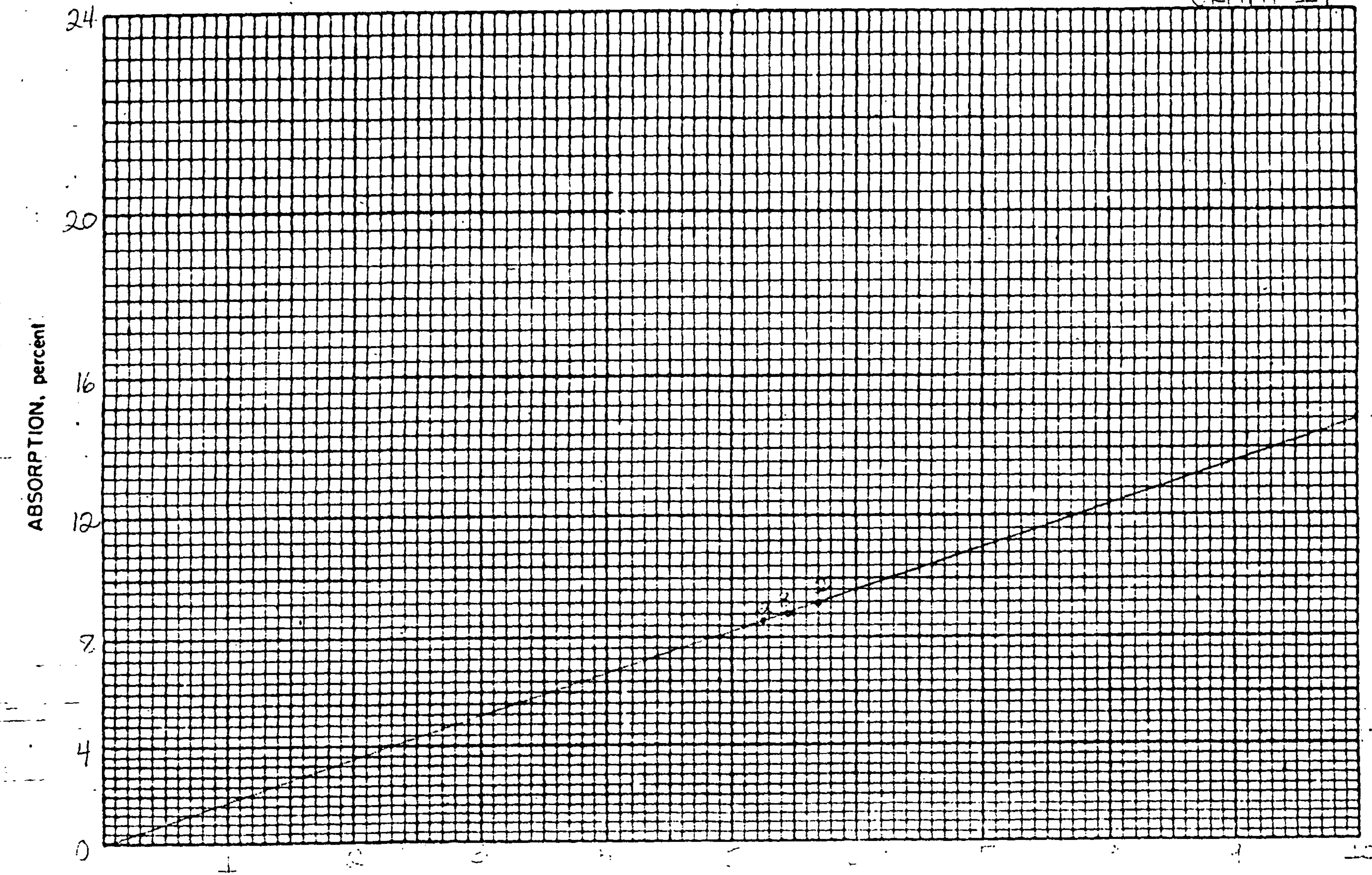
Unknown sample analyses

Table XVIIb

Sample No.	Dilution Factor	A.A. Reading	%Abs	PPM	PPM Orig. Solution
GE 12SN 01 #2	5	.042	9.2	5.7	28.5
" 11SN 01 #8	5	.040	8.8	5.45	27.25
" " " #12	5	.039	8.6	5.25	26.25

ANALYSES OF GE 12 ON OL #2, #8, AND #10

GRAPH 17



Data for Graph XVIII

K Analysis of plates S_1, S_2, and S_3

Calibration curve for K

Table XVIIIa

PPM	A.A. Reading	%Abs
1	.024	5.4
2	.0488	10.6
4	.1066	21.8
6	.1734	32.9
8	.2436	42.9
	.3202	52.2

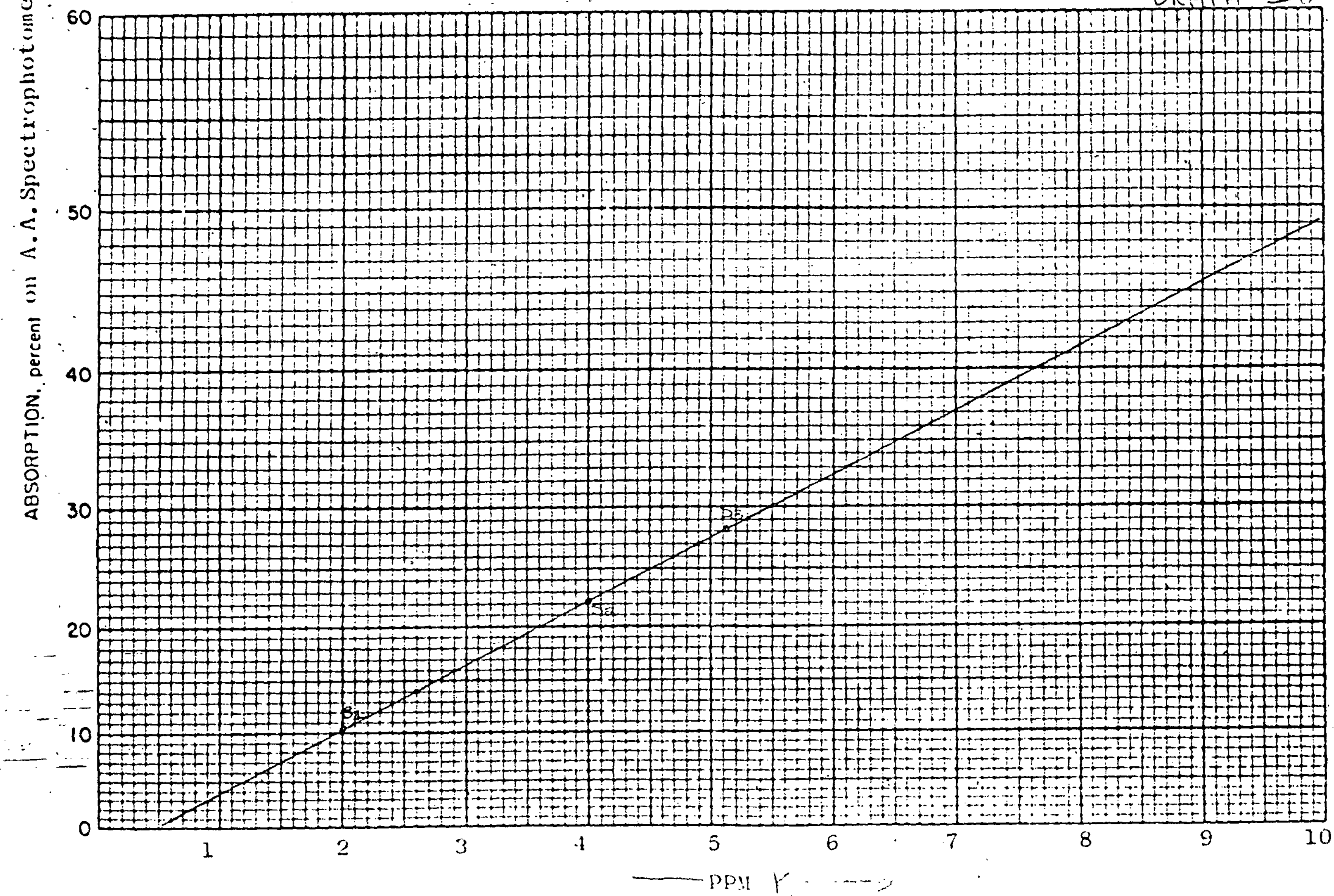
Unknown sample analyses

Table XVIIIb

Sample No.	Dilution Factor	A.A. Reading	%Abs	PPM	PPM Orig. Solution
S_1	0	.0492	10.7	2.0	2.0
S_2	0	.107	21.8	4.0	2.0
S_3	2	.1458	28.5	5.15	10.30

ANALYSES OF SAMPLES P.O.T. S_1, S_2, S_3.

GRAPH 18



Data for Graph XIX

Calibration curve for K

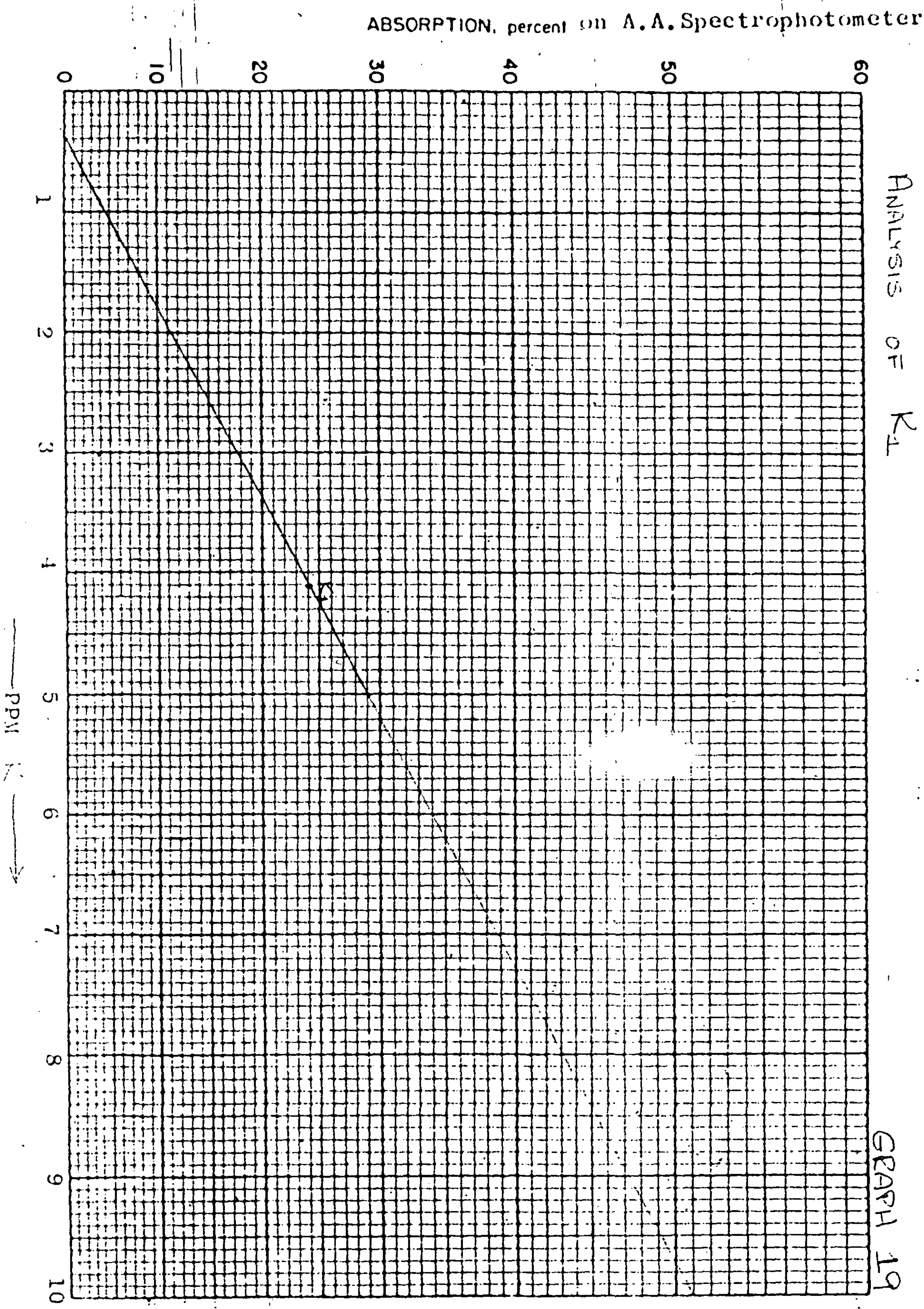
Table XIXa

PPM	A.A. Reading	%Abs
1	.028	6.30
2	.063	13.5
4	.108	22.1
6	.178	33.7
8	.257	44.6
10	.333	53.6

Unknown sample analyses

Table XIXb

Sample No.	Dilution Factor	A.A. Reading	%Abs	PPM	PPM Orig. Solution
K_1	250	.114	23.1	4.1	1025.0


ABSORPTION, percent on A.A. Spectrophotometer
0 10 20 30 40 50 60
ANALYSIS OF K+
GRAPH 19
1 2 3 4 5 6 7 8 9 10
PPM K

Data for Graph XX

K analyses of plates S_1, S_2, S_3

Calibration curve for K

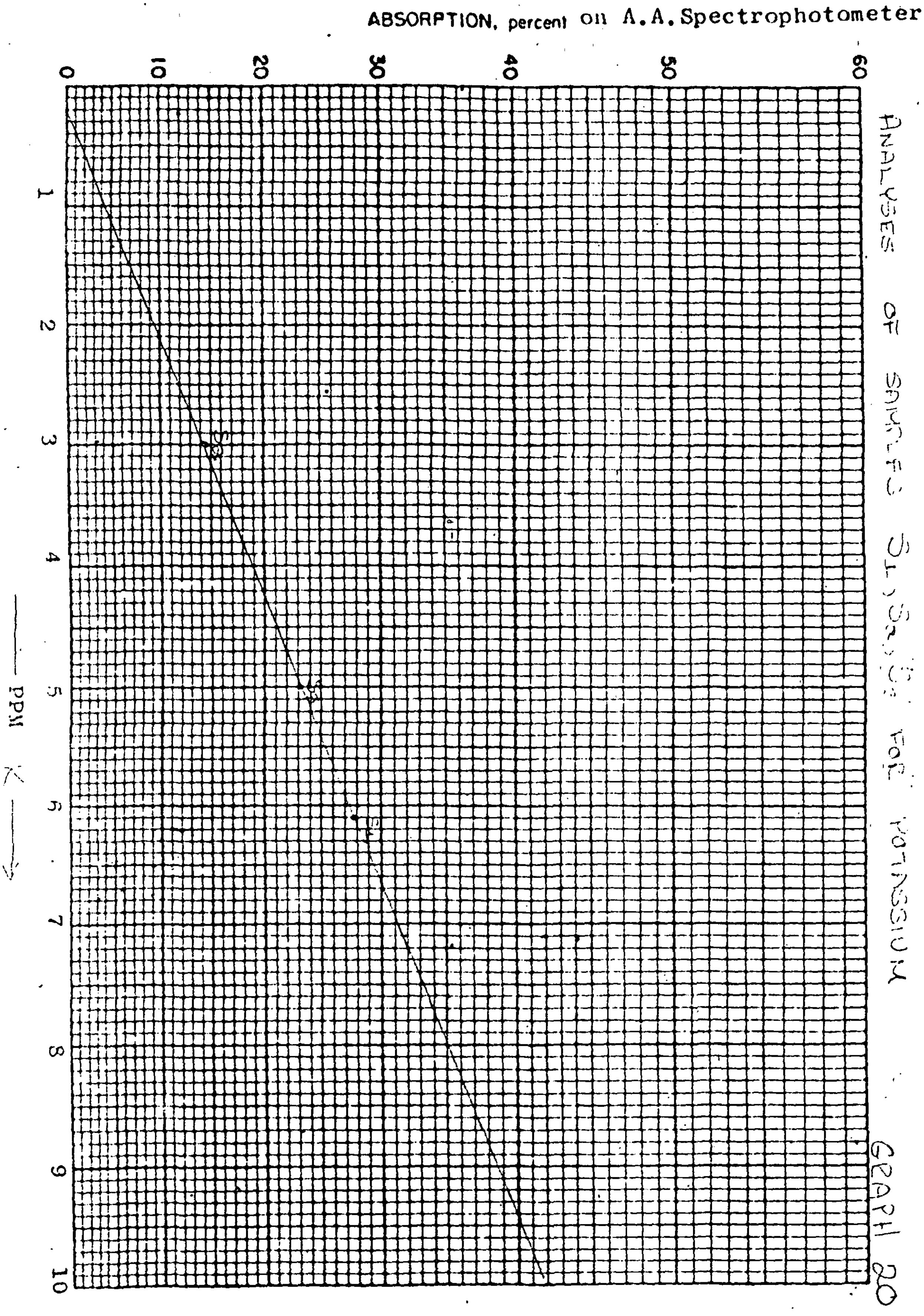
Table XXa

PPM	A. A. Reading	%Abs
1	.0138	3.1
2	.0262	5.8
3	.0450	9.8
4	.0666	14.2
5	.0930	19.3
6	.199	24.0
7	.150	29.2
8	.176	33.3
9	.208	38.1
10	.234	41.8

Unknown sample analyses

Table XXb

Sample No.	Dilution Factor	A. A. Reading	%Abs	PPM	PPM Orig. Solution
S_1	10	.121	24.3	6.1	61.0
S_2	"	.048	10.4	3.0	30.0
S_3	"	.093	19.3	5.0	50.0


ABSORPTION, percent on A.A. Spectrophotometer
0
10
20
30
40
50
60
ANALYSES OF SAMPLES
FOR POTASSIUM
GRAPH 20
1
2
3
4
5
6
7
8
9
10
PPM K

EXPERIMENTAL - PART 3

DESIGN VARIABLE CELL ANALYSES

TABLE XXI

Design Variable Cells 12 AH GE

Group No.	Ser. No. of Cell	Pack No.	No. of Cycles	Plate Thickness (mm) Pos.	Plate Thickness (mm) Neg.	Plate Weight Pos.	Plate Weight Neg.
1	004		None	0.72	0.79	13.69	15.46
1	001	3D	5833	0.74	0.80	13.97	14.83
2	004		None	0.72	0.80	13.85	15.87
2	001	3E	5841	0.74	0.79	14.00	14.87
4	001		None	0.68	0.79	13.02	14.71
4	002	3G	5844	0.72	0.79	13.31	13.83
5	001		None	0.74	0.79	13.32	15.43
5	002	3H	5840	0.77	0.80	13.65	14.92
6	002		None	0.72	0.79	13.65	15.59
7	005		None	0.91	0.74	15.34	14.13
8	002		None	0.90	0.71	15.35	14.02

TABLE XXII

Design Variable Cells 12 AH GE

Group Serial No.	Chemical Capacity (Amp-hr) Pos.	Neg.	Electrochem Capacity (Amp-hr) Pos.	Neg.	OH^- (meq)	CO_3^{2-} (meq)	% $Co(OH)_2$ (in Pos.)
1-004	22.64	34.02	14.24	21.69	199.8	69.06	2.81
1-001	21.22	30.03	15.72	17.40	232.8	53.4	2.76
2-004	21.74	36.28	16.77	23.86	294.1	57.8	3.77
2-001	22.90	30.77	16.17	17.89	293.8	65.3	2.98
4-001	20.02	30.48	14.28	21.75	264.5	54.9	3.12
4-002	21.44	26.17	13.58	14.27	219.2	42.7	2.70
5-001	22.69	34.65	17.24	24.99	230.9	66.2	3.20
5-002	22.44	32.11	16.72	22.22	213.5	77.0	3.06
6-002	22.36	36.62			237.8	48.3	3.23
7-005	25.23	32.54			198.2	97.6	3.57
8-002	25.63	32.93			203.6	109.8	3.97